Forming of steel polymer laminates.

Bruton, Paul

Forming of Steel Polymer Laminates

By

Paul Bruton BEng(Hons)

Thesis submitted to the

University of Wales

For the Degree of

Engineering Doctorate

Department of Materials Engineering

University of Wales Swansea

March 2001

Academic Supervisors: Professor JD Parker
Dr C Arnold

Industrial Supervisor: B Bastable

DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

Signed .....(candidate)
Date 15 June 2001

STATEMENT 1

This thesis is the result of my own investigations, except where otherwise stated. Other sources are acknowledged by footnotes giving explicit references. A bibliography is appended.

Signed .....(candidate)
Date 15 June 2001

STATEMENT 2

I hereby give consent for my thesis, if accepted, to be available for photocopying and for inter-library loan, and for the title and summary to be made available to outside organisations.

Signed .....(candidate)
Date 15 June 2001

I hereby give consent for my thesis, if accepted, to be available for photocopying and for inter-library loan **after expiry of a bar on access approved by the University of Wales on the special recommendation of the Constituent/Associated Institution.**

Signed .....(Candidate)
Date 15 June 2001

Acknowledgements

Firstly, I would like to thank my wife Cathy, without her love, encouragement and understanding this may never have been completed. I also extend my gratitude to my erudite supervisors Professor Jonathan Parker, Dr Cris Arnold and Brian Bastable. Their wisdom, experience and advice kept me motivated over a long four years. I also value the support and direction given by my Theme Champions Dr Mick May and Dr Chris Elliot. Acknowledgement also goes to everyone, past and present, in the Packaging Theme Group, whose positive participation in my presentations helped advancement of this project.

Special mention must also go to the film laminate team, John, Sarah, Helen, Cerith and Jago; their assistance, advice and drinking abilities will always be remembered. I am indebted to everyone else within the Engineering Doctorate Scheme, Corus Group and EPSRC, who assisted me in my project, especially members of the former Tinplate R&D department. Finally, I need to express my heartfelt thanks to all of my family and friends, for their social and moral support, it is always appreciated.

As someone very dear to me keeps saying:

"Isn't it time you stopped draining the honest taxpayers resources and got yourself a real job?"

Maybe, dad, just maybe.

Project Summary

Steel polymer laminates are a growing sector within the packaging industry. They typically consist of a chrome passivated steel substrate, between 0.15 and 0.40mm in thickness, which has a polymeric film laminated to each side of the steel base. For most products the polymer layer is either polyethylene terephthalate (PET) or polypropylene (PP) of between 15 and 200μm thickness. Applications for this product include aerosol caps and domes, food canisters and beverage containers.

The current programme has investigated two methods of forming film laminated materials; deep drawing and biaxial stressing. The first area investigated drawing of a cup from a flat disc used a range of steel polymer laminate and tooling variables. Analysis of a broad spectrum of laminate products showed that the steel substrate has a much greater influence on cup height than polymer film specification. Of the variables considered, cut edge and punch diameter had the most profound effect on cup dimensions. Factors including blank holder force and die radii were also shown to influence forming. For one laminate family a predictive equation for cup height has been developed. This has proved to be robust for a range of formed components.

The second area of investigation, biaxial stressing, was designed to develop an understanding of the stress whitening observed on the domes of certain beverage cans. This defect is a weakened area of the coated material and has potential to cause failure of a filled component. A laboratory testing procedure was developed, which allowed this problem to be studied under closely controlled conditions. Subsequent analysis has shown that the rolling lubricant applied to the substrate prior to film lamination causes the problem. These lubricants are shown to have a solubility parameter close to that of the stress whitened coating. The mechanism of occurrence is shown to be a combination of laminate age and environmental stress cracking. Complete elimination of either the biaxial stress or active media is shown to be impracticable due to component specification and standard industry working practice. Two successful preventative methods were found; forming of the component before the laminate had aged to a point where crazing occurs, or heating the component above the polymer glass transition temperature prior to deformation.

Notation

ATBC	Acetyl tributyl citrate oil
BA	Batch annealed
BHF	Blank holder force
BHP	Blank holder pressure
BSO	Butyl sterate oil
CA	Continuously annealed
C_{ed}	Cut edge diameter
DOS	Dioxysebacate oil
D_{dia}	Internal die diameter
DR	Double reduced (steel substrate)
D_{rad}	Die radius
D_{rat}	Draw ratio
DRD	Draw redraw (can making process)
DSC	Differential scanning calorimetry
DSRD	Draw stretch-redraw (can making process)
DTR	Draw thin-redraw (can making process)
DWI	Draw and wall ironed (can making process)
σ	Stress
Δh	Cup wall earing measurement
Δr	Planar anisotropy value
ECCS	Electro chromium coated steel
ESC	Environmental stress cracking
ETL	Electrolytic tinning line
ε_t	Thickness true strain
ε_w	Width true strain
$\dot{\varepsilon}$	Strain rate
k	A constant
LD_{rat}	Limiting draw ratio
L_f	Final length
L_i	Initial length

m	Strain rate sensitivity value
n	Work hardening exponent
NMI	Non-metallic inclusion
P_{dia}	External punch diameter
PEI	Polyethylene isophthalate
PET	Polyethylene terephthalate
PP	Polypropylene
PPN	Polypropylene with nylon top-coat
P_{rad}	Punch nose radius
PVC	Polyvinyl chloride
RD	Rolling direction
r	Plastic strain ratio (r-value)
$r_m \quad (\bar{r})$	Average plastic strain ratio
S_{st}	Substrate ultimate tensile strength (MPa)
S_{th}	Substrate gauge (mm)
T_f	Final thickness
TFS	Tin free steel (an early name for ECCS)
T_g	Glass transition temperature
T_i	Initial thickness
T_m	Melting point
T_x	Recrystallisation temperature
VOC	Volatile organic compound
W_f	Final width
W_i	Initial width

1 Introduction

The packaging market is a substantial part of the global economy, with world wide turnover reported at $550bn for 1998 and expected to grow four-fold in the next decade. Film laminated steels are a growing sector within the packaging industry, with over 15,000 tonnes manufactured in Britain in 1999. They typically consist of a steel substrate, between 0.1 and 0.4mm in thickness, which has a polymeric film applied to each side of the steel base. The polymers used for packaging applications are either polypropylene or polyethylene terephthalate, with a thickness of between 15 and 200μm. The steel polymer lamination process applies the semi-crystalline film to the steel substrate and then heats the laminate material to the temperature required to produce an amorphous polymeric structure with good interface adhesion. Finally, the laminate is rapidly quenched to ambient temperatures; this retains the coatings amorphous structure. Film laminate materials have many applications including aerosol cups and domes, paint canisters, food cans and more recently, beverage containers. Non packaging uses include white household goods, reflective strip light backing and cable wrap.

For a material with so many end uses, little is known about the laminate's forming properties. It was the intention of this programme to investigate how the mode of deformation influences the forming characteristics of film laminated materials. This is to be split into the two main areas of can making; deep drawing, for production of the shell, and biaxial stressing, for manufacture of the dome on beverage cans.

Deep drawing of shells, or cups, from metal substrates, is an area with a moderate amount of published data. However, only limited publications consider thin gauge materials used in packaging applications. It was therefore intended to investigate how film laminate materials behave under a single stage operation from a flat disc to a drawn shell. The experiments were sub-grouped into three main areas; effect of steel substrate, effect of polymeric coating and mechanical tooling issues. Each of these sections was then broken down into its constituent elements for ease of analysis and discussion. By more fully understanding each of these factors influencing drawing of

film laminated materials, it was hoped to find optimum specification for a given end application.

The second area of investigation centres around biaxial stressing of film laminated materials, analogous to production of a dome in can making. It has been noted that in production of some commercial cans, a white ring is visible on the internal clear coating of the domed region of the container. Therefore, it was intended to investigate this phenomenon, establish the factors affecting this occurrence and suggest methods of elimination or prevention.

These two areas of investigation will then provide an insight into factors influencing the forming of film laminated materials, their relative importance and their effect on final deep drawn container properties. This can then be used by producers and manufacturers of film laminated materials to provide optimum packaging containers for a variety of end uses.

2 Literature Survey

2.1 Materials Developments

Throughout time man has been trying to develop ways in which to make food last longer, and yet remain transportable. The earliest types of food and drink containers were animal skins, and with evolution, earthenware bottles and then glass jugs were developed. In 1795 the French government, realising the imminence of the Napoleonic wars, offered a prize of 12,000 FFr. for the discovery of a practical method of food preservation.[1] A chronology of key developments in food and drink container packaging history is shown in Figure 2.1. This summarises the information presented in section 2.1.

2.1.1 Containers Engineering Evolution

Nicholas Appert, a French confectioner, developed a method of sterilisation to claim the French government prize.[2] With this system, a filled glass container was submerged in boiling water, extending the life of the contents. This prompted others in Europe to investigate similar areas and in 1810 King George II of England granted Peter Durand a patent for a tinplated iron can for use as a food container.[3] The early tinplate containers were three piece, hand soldered and riveted cans. However, production speeds were low and even the best craftsmen could only produce about sixty cans per day. In 1846 Henry Evans invented a device for making cans in a single operation, increasing production speeds to about one per minute.[3] By 1880, an automated can making machine had been introduced in Britain, allowing canned foods to become a growth industry. As the industrial revolution drove forward, machinery continued to be developed, production times and costs decreased and the popularity of canned foodstuffs expanded.

At the end of the Nineteenth century, tinplated cans were mainly used for tinned fruit and vegetables.[4] Drinks at this time were being kept in glass bottles with a cork as a seal. Closures for glass containers of carbonated drinks progressed from the marble

'codswallop' stopper through to the 'lightning', Grolsch type, resealable top. These methods were expensive and difficult to clean, creating a need for a cheap quick capping innovation. In 1891, William Painter developed the 'crown cork seal', which combined an air tight top with an easily removable lid.[5] However, glass containers of the time were relatively expensive and heavy, their shape and size variability was high, and they were refillable requiring a returnable deposit upon purchase.

In 1935, tinplate was first used in the drinks industry as a beer can, at the Felinfoel brewery in Llanelli, South Wales.[6,7] The container was a simple three-piece can, shaped like a bottle, with a crown cork seal. This shape had the advantage of being compatible with the filling machinery of that time. These cans however were difficult to stack; a factor, which led to the development of the 'flat top', can a few years later. It was soon realised that beers were very sensitive to metallic contamination.[8] A tinplated can would rapidly modify the taste of the alcoholic contents necessitating the need for an internal protective barrier coating. A variety of linings were examined including vinyl coploymers and waxes.[9] These new linings improved the short-term life of canned beers, but an effective long-life lacquer was still to be developed.

With the outbreak of the Second World War came a reversal of packaging fortunes. Label sizes, were reduced to a minimum, to save on raw materials and public canned beer consumption was halted under rationing. The need for metal to build artillery and munitions caused tins to be replaced by card boxes and metal caps by corks.[4] Beer rationing was finally ended in 1953 and the flat top beer can became popular. However, they were difficult to open requiring a special triangular opener.

The 1950's brought many breakthroughs that affected the packaging industry. The basic substrate used for tinplated cans began to change. Early tinplate consisted of iron hammered into flat sheets and dipped into molten tin baths. Expanded demand caused new technological developments in the form of an electrolytic tinning line, which coated the strip by electro-deposition. The strip was passed through an electrolytic solution, which acted as the anode, depositing the tin on the cathodically charge strip steel. This method gave much more consistency than the old hot dipping method and used substantially less tin.[10] It also allowed different coating weights to be applied to different sides of the steel strip, allowing lower coating wastage and

minimum over specification. This gave tinplated steel a monopoly in the three-piece can making market because aluminium could not be seam welded due its low electrical resistance

The development of television and associated advertising ensured striking advances in printing and packaging. These changes resulted in product differentiation and branding. Carbonated soft drinks at this time became popular with the new 'teenagers' and the likes of Pepsi and Coca-Cola were produced in glass bottles with the by now universal metal crimped top.

In 1963 the Aluminium industry devised a new easy to open beverage can using a patented ring-pull. Within months almost all cans had incorporated this new innovation and the standard easy open end became commonplace. The following year saw possibly the most important technological breakthrough in canmaking history. Reynolds Metal Co. developed a new two-piece forming process; the draw and wall ironing (DWI) technique, for the manufacture of can bodies.[1,2] This new process required no seam welding, thus breaking the steel monopoly and allowing aluminium into the marketplace. The new all aluminium beverage can was rapidly developed and marketed. It gave a brighter, shinier finish, which enhanced product differentiation and increased market share. Reynolds Metal Co. made a further development, introducing a 12-oz necked-in aluminium can which reduced the standard 2"11/16 (211) diameter end to a 2"9/16 (209). This new technology used a spin flanger instead of the usual die flanger, reducing the metal stresses, thus combating the previous problems associated with cracked flanges.

In response to the new shinier finish aluminium can, the steel producers developed a double reduced (DR) tinplate grade. This new grade, discussed more fully in **2.2.4**, was lighter and stronger than the previous single reduced (SR) grades. The DR tinplate also gave a shinier, brighter finish than its SR counterpart, helping the steel industry compete and slow the growth in all aluminium can sales. The new aluminium can was lighter, quicker and cheaper to produce than the old steel three-piece can. However, if the new two-piece DWI technology was used on a steel substrate, then new developments were possible. Steel possesses one major advantage over aluminium in that it significantly work hardens upon ironing of the cup wall. This

phenomenon of work hardening allows the possibility of downgauging, since a much thinner substrate can produce a similar strength to the traditional thickness. Any lightweighting has benefits in that it reduces the cost of the can since material costs constitute approximately sixty percent of the price of an empty (steel) can.[11] Furthermore its lower weight makes it cheaper to transport. Tinplate also had the benefit of needing less lubricant than aluminium, since the tin coating itself acts as friction reducer by facilitating the wall ironing process.

Tin prices were very high around this time and, on occasions, in short supply.[10] To stop aluminium making further inroads into the coated steel market, a new product was developed, originally called tin free steel (TFS), now more commonly electro-coated chromium steel (ECCS).[12] It was initially developed by the Japanese as a product called Hi-Top, consisting of a duplex coating of metallic chromium and chromium sesquioxide. The new process had many advantages over the traditional tinplate coating. It required substantially less coating weight compared to the best electrolytic tinning line achievement at that time.[10] The coating also had superior lacquer adhesion than the tinplated steel. However, it did have one major disadvantage in that it could not be soldered, three-piece cans using this coating had to be seam welded.

In 1969, Reynolds Metals Co. introduced a new harder temper product from the same previously used alloys, allowing further lightweighting of the aluminium can. Throughout the next decade ever further lightweighting occurred, in a continual but gradual improvement process. However, when lightweighting a component a conflict of interests occurs; to achieve the required wall and axial crush strengths at a thinner gauge, stronger base material must be used. This reduces the ductility of the metal, lowering the cup height-diameter draw ratio, necessitating extra drawing steps to achieve the same can wall height. A situation of diminishing returns becomes obvious; the substrate is more expensive, less of it is required, but the can is lighter, thus only resulting in small overall cost improvements.

It can be proven, that steel has more room for downgauging than aluminium, because the average steel grade has four times the strength of a similar thickness aluminium grade.[13] Steel generally, is heavier than aluminium and as such will always be at a

weight disadvantage. However, steel tends to be cheaper, providing can makers with an interesting dilemma, whether to go for lowest cost or lightest can.

By the end of the 1970's, two other two piece can making processes had become available, both of which are discussed more fully in **2.3.2**. The first was the draw-redraw process DRD, which differs from the DWI process because no wall ironing takes place. The first stage, cupping, remained the same, but the second was where the difference occurred. The cup is redrawn in successive operations through progressively smaller dies until the required dimensions are achieved. This process has the advantages of needing less lubricant than its DWI alternative, and it also protects any coating more effectively. The second stage was the draw-thin redraw process (DTR), which consisted of a redraw process with a small amount of wall thinning midway through the forming sequence.

In 1980, two-piece cans dominated the beverage market, being faster and cheaper to produce than their three-piece counterpart. Of the beer can total, 75% were two piece aluminium, 20% were two piece steel and the remaining 5% were three piece steel.[13] Significant developments originating in the USA were also introduced. The first was the retained ring-pull, or stay on top (SOT), which negated the disposal and subsequent litter problems of the old ring-pull. The second was a necking phase at the top of the carbonated soft drinks can, whereby a new 2"6/16 (206) diameter neck was introduced instead of the straight walled 2"9/16 (209) standard diameter can. This reduced the amount of material needed for the lid by approximately 14%; the extra cost of producing this neck was more than offset by the lower cost of making the new smaller lid. Over the next decade can ends were reduced from the necked-in 206 ends to 204, and even some 202 diameters. This again provided can manufacturers with a significant material saving in the costly can top. Further size reductions were, and remain, theoretically possible, but the aperture through which the consumers would drink the liquid, must be either redesigned, or reduced in size.

All cans produced at this time were either all aluminium, or a steel body with an aluminium end. This provided some recycling difficulties, but offered a chance for increased steel usage in the form of an all steel can. In 1992, the three major European tinplate producers, British Steel Tinplate, Hoogovens and Rasselstein announced

plans to develop an all steel 'ultimate can'.[14] This project planned to use the steel Ecotop, on a smaller 202 neck diameter, and a starting gauge of 0.22mm, instead of the usual 0.27mm. The consortium, predicted up to a 30% reduction in the weight of this can, within two years. The first all steel Ecotop can was launched by Sainsbury's on two products in 1993.[15] Initial sales figures were promising, but the can was withdrawn from the shelves in 1996, following a steady drop in demand.[16]

After the huge success of the traditional, shaped PET 12-oz Coke bottle. Coca-Cola, worried about declining market share, initiated the concept of the shaped can.[17] Three-piece steel shaped cans were test marketed in Germany and South East Asia in 1994/5, with a growth in sales, following increased consumer demand. This prompted Coca-Cola to introduce a trial run in the U.S.A. of two-piece aluminium DWI contoured cans, shaped to the 'signature pattern' of the traditional bottle.[18] A recent report,[19] highlighted that the contour can could be lighter than previously thought, using a combination of finite element analysis and new hydraulic or pneumatic forming processes. However the contour can also has many disadvantages. It is, at present, a slow can making process with the best lines running at approximately 600 cpm (cans per minute),[20] compared to a conventional high-speed can making line of 1500 cpm.[21] Also, as a result of the shaping, the can is more expensive; Coca-Cola have announced that they will initially bear the brunt of the excess costs, to ensure product differentiation.[17] It remains to be seen if the consumer will pay for the new technology in the long run.

More recently British Steel Tinplate and Redicon, announced the combination of the Ferrolite material, with a new shaping process, named the RBS project, to produce a new laminated shaped can. It has been predicted that this can offers both cost and strength advantages over the aluminium DWI can, presently used by Coca-Cola in their contoured can trial.[22] Time will tell if either of these shaping developments prove profitable.

Concurrent to all these developments, fundamental changes were taking place with the steel substrate used for packaging applications. There were many process developments that resulted in better chemistry control, which reduced the number of

defects produced in thin gauge material. These improvements in the manufacture and control of steel production are well documented in other sources.[23, 24]

2.1.2 Coatings Development

The earliest effective internal coatings used for food contact uses were lacquers, mostly oleo-resins. These resins were sprayed onto the 'inside' of the can before seaming. After joining, additional lacquer was applied to the soldered seam, then finally the whole can was sprayed with a vinyl resin. This process produced a can, which significantly enhanced the life of its contents, typically iron pick-up levels were now quoted at 0.3ppm over a six month period.[9] Oleo-resins did have one major disadvantage; they had a loose macromolecular structure and were therefore slightly permeable. This caused any foods containing sulphur to react with the can wall producing harmless, but unsightly, stannous sulphides. To overcome this problem, white zinc oxide powder was added to the lacquer, which preferentially reacted with the sulphur to produce another white compound; this was not visible on the internal walls of the can.[25]

The DWI process caused some internal coating problems. The standard oleo-resin lacquer had adhesion problems during deep drawing. As a result, a new epoxy phenolic resin was developed which, once hardened, possessed a tough impermeable layer whilst retaining excellent adhesion characteristics. This new lacquer reduced iron pick-up levels in an electro-deposited tinplate can, over a six month shelf-life, to below 0.3ppm (parts per million). Epoxy phenolic resins have some disadvantages. Firstly they have a high volatile organic compound (VOC) content, which helps to sustain hazardous ground-level ozone.[26] Secondly, these lacquers are difficult to recover and recycle, due to their complex structure. These drawbacks were not investigated and fully understood until a few decades later.

Concerns about the safety of VOC's, coupled with the possibility of dioxin and PCB generation from incineration of PVC and vinyl based lacquers,[13,27] caused canmakers and coatings producers to consider water-borne protective lacquers. These new lacquers dispensed with the need for organic solvents by replacing them with water, but this system did have its drawbacks. Firstly, the adhesion and flow properties of the

lacquers were poor, requiring the addition of a co-solvent to alleviate these problems.[28] Secondly, in comparison to the solvent based lacquers, the water-based systems required an extra drying stage after application, increasing production costs and decreasing line speed. The other popular alternative was to increase the solids content within the lacquer, thus requiring less solvent to apply the same quantity of coating. This concept also had a disadvantage; as the level of solvent was reduced, the viscosity of the lacquer increased, soon causing application difficulties to outweigh any savings produced from reducing the solvent content.[29] The possibility of legislation restricting VOC content continues to drive coatings manufacturers to improve and update lacquer technology.

In the late 1980's, the next important technological breakthrough occurred; polymer coated steel laminates were produced. Two companies developed methods of manufacturing these film laminates, Toyo Kohan and Carnaud Metal Box. The first method, developed by Toyo Kohan uses a biaxially oriented Polyethylene Terephthalate film. This film is applied to an ECCS substrate preheated to above the polymer melting point and then rapidly cooled. The laminate's polymer coating has two distinct structures. Nearer to the substrate the polymer is amorphous, whilst the top remains crystalline. This results in good adhesion and excellent barrier properties.[30] The second method, developed by Carnaud Metal Box, differs from the above method in that a bonding layer is used to achieve the necessary adhesion levels. This allows dissimilar polymers to be applied simultaneously, since little or no substrate preheat is required, producing wider possible ranges of applications. The latter product, called Metpolam, from metal-polymer-laminate, is an ECCS grade steel coated in either Polyethylene Terephthalate (PET), or Polypropylene (PP); a nylon coating was also patented, but rarely used.[31] The polymer coating became available in white and clear versions, with the possibility of other colours upon request. This product was subsequently licensed from CMB by British Steel Tinplate in 1988, and in 1992 the product was renamed 'Ferrolite'. The precise production process of this product is discussed in section **2.2.6**.

A polymer coating of the Ferrolite type has many advantages over a conventional lacquered can. It is produced in a single pass, unlike the two or three passes required for sheet lacquering.[32] The solvents are virtually eliminated because no PVC coating

is required and therefore volatile organic compound (VOC) emissions are minimised. Ferrolite can also be recycled directly into the steelmaking plant without adverse effects on emissions or steel quality.[33] Using Ferrolite for the canmaking process requires much less lubrication than any other method because the coating itself acts a lubricant, but yet still stays in intimate contact with the substrate, without affecting the coating performance. Polymer coated products, especially the white versions, have a strong aesthetic appeal, appearing clean and hygienic. They are also much easier to print than the standard lacquered can, with the coloured PET or PP coating providing a strong backdrop for any subsequent decoration and/or printing.

2.1.3 The Perfect Package

All these developments assist in the quest towards the ultimate container. The ideal properties of a perfect drink container can be broadly classified under five main headings.

Production costs: these need to be kept to a minimum therefore requiring cheap abundant raw materials, fast production speeds, low plant costs and minimal energy usage. Consideration of costs is paramount in persuading fillers to use a particular product.

Durability: there is a need to consider dent resistance, container strength (both full and empty), resistance to chemical attack and tampering. Additionally, the package should be easy to transport.

Consumer and retailer requirements: the retailer requires a shape, which minimises space and has strength to withstand stacking. With regard to the consumer, aesthetics, ease of opening and resealability are all important considerations in persuading consumers to buy a product; this is possibly the most important factor, because without sales the product will never succeed.

Lowest container weight: the use of lighter, stronger raw materials, thus requiring less in each package. The lower weight usually has a double benefit, because each unit container is lighter it is cheaper to transport.

Environmental considerations: new legislation is requiring the packaging sector to produce a more environmentally friendly product. This legislation and other parallel issues consider recycling capability, biodegradability and the reduction of potentially harmful emissions.

2.2 Production & Processing

The following production sequence maps typical Corus Packaging Plus products from entry to the tinplate works to exit as a finished coil.

2.2.1 Steelmaking

Tinplate designated material following standard ironmaking and steelmaking production routes is described in detail in other publications.[34,35] Molten steel enters the secondary steelmaking vacuum degassing unit. This unit maximises uniformity of both the composition and temperature of the steel, prior to casting. A typical composition range for tinplate made from unalloyed low-carbon mild steel is indicated in Table 2.1.

Compositional qualities

Carbon increases strength and hardness, thus reducing both elongation and ductility.
Sulphur reduces ductility and corrosion resistance whilst degrading surface quality. Free sulphur in a melt reacts together with iron to form iron sulphide (FeS), this is a very brittle compound which greatly reduces the ductility of the resultant steel.[36] Sulphur removal, in the form of ladle desulphurisation, is achieved prior to entry to the steelmaking vessel, a process that considerably reduces the sulphur content in the molten metal. Combined with manganese additions this system allows sulphur levels to be kept within working limits. Large inclusions can lead to catastrophic failure in very thin steel, jamming cupping presses and causing extensive tooling damage, which results in lost time costs. As such, removal of large inclusions in material destined for can making is paramount to provide useable, high quality material
Phosphorus removal has conflicting requirement properties with sulphur removal in the steelmaking vessel. Phosphorus does cause a reduction in ductility by entering the ferrite phase of the steel as a solid solution.[36] However, it only has a marginal adverse effect on corrosion resistance and, in preference to sulphur, some residual phosphorous is always present in a steel composition.
Copper, at the low levels encountered in tinplate compositions, has very little effect on strength and ductility, although it does benefit atmospheric corrosion resistance.

Manganese is primarily added to the steel to combat the undesirable effects produced by sulphur. Manganese and sulphur react together to preferentially form manganese sulphide, which remains in the steel causing slight pinning effects. Such adverse properties of manganese sulphide are however preferable to those of free sulphur.
Silicon content is mainly derived from residuals in the ironmaking process, it causes a slight hardening effect by entering as a solid solution into the ferrite grains.[36] Silicon also adversely changes the corrosion properties of the steel.
Nickel, at low levels has very little effect on the steel's mechanical properties. However, it can hinder the formation of the iron/tin interfacial alloy during the tinning process.
Nitrogen is a very good strengthening element. Very small additions can greatly increase the strength of the steel. At all times nitrogen levels need to be closely controlled.

2.2.2 Primary Cold Reduction

The hot rolling process produces a layer of oxides, or scale, on the surface of the sheet. This scale must be removed before any further deformation can occur. The iron oxides are generally removed by pickling, a continuous process, where coils are welded, head to tail and fed through a warm acid bath; hydrochloric, sulphuric or most recently hydrofluoric acids are used. Under pickling and over-pickling both adversely affect surface quality, the former causing rolled in defects and the latter etch pits. Additionally, a mechanical scale breaker may be employed to facilitate the pickling process. Before exiting this stage, the coil must be fully rinsed and dried, to prevent any downstream contamination.

Cold rolling is the reduction of strip thickness below the recrystallisation temperature. The percentage reduction typically used in tinplate steel is in the region of 85-90%. Reducing the gauge from about 2.0 to 0.2mm. Tandem mills, are usually used for cold rolling. Five or six four-high mills are common, although the last mill stage may be of a six-high configuration allowing extra thickness reduction and gauge profile accuracy. Due to the high reduction in thickness and low rolling temperatures involved, a phenomenon of work hardening occurs, i.e. the reduction in thickness is associated with an increase in the strip strength. The coil speeds also increase at each

stand, a factor which contributes to temperature rises through the mill; this can be anything up to about 200°C, mainly due to frictional heating effects. Work hardening occurs, because of the lower process temperature, high-induced strains and the body centred cubic (BCC) nature of the steel. As a greater percentage reduction is applied, the strip gets harder, necessitating the majority of the thickness reduction to occur in the first few stands whilst the steel is relatively softer. This allows the latter stands to concentrate on strip steel's profile and shape.

The coil, upon exit from the cold mill, has a cold worked grain structure and is contaminated with rolling lubricant. Both of these must be removed before any further processing is to occur. To remove the lubricants and any coolant, the coil must be passed through a series of electrolytic solutions. After leaving these tanks, the coil must be rinsed, and then dried, using a hot air blower.

2.2.3 Annealing

The heavily cold worked structure, caused by the large reductions in thickness, gives the steel a very high strength and low ductility, a disadvantageous property for deep drawn tinplate applications. Annealing is a process where the steel is heated to a sub-critical region of the iron-carbon phase diagram, allowing the ferrite to recrystallise, from cold worked elongated to fully recrystallised equiaxed grains. There are two, very distinct, types of annealing:
Batch annealing, and
Continuous annealing.

Batch annealing

Batch annealing (BA) is a process where coils are placed in a sealed box, with a controlled atmosphere, and subsequently heated up to about 650°C for 5-12 hrs, then furnace cooled to a required heat treatment cycle. A typical batch annealing cycle is indicated in Figure 2.2.The box can contain up to four stacks, each three to four coils high. The atmosphere within the unit is either 100% N_2, or 95% N_2 and 5% H_2 (HNX). These conditions help prevent surface oxidation, which can lead to sticking of adjacent laps within a coil. To prevent distortion the heating and cooling cycles must

be closely controlled so that the strip shape is satisfactory for further processing and meets customer requirements. The total process time for BA products can be in excess of one week.

The BA route does have some disadvantages. Firstly, there are large temperature gradients, both across and up the furnace. These gradients can be anything up to 40°C, causing variable grain sizes and subsequent mechanical properties, producing variations in the consistency of deep drawn characteristics. Secondly, this process can cause sticking between adjacent laps of the coil, which result in unacceptable surface quality for many thin gauge products. Thirdly, the whole process is very time consuming, causing a bottleneck in throughput, and reducing the efficiency of the whole steelmaking process. The BA process however, if logistics are correctly used, can anneal different coils of different thickness' at the same time, advantageously using the temperature gradients present within the unit. Different coils need different cycles, so if the temperature is accurately known, the variations can be effectively used.

Continuous annealing

To overcome the time constraints and inconsistency of the BA process, a continuous annealing (CA) system was developed. Here the coils are welded, head to tail, and heated to a high temperature for a very short time (~700°C for 6 to 24 seconds), then cooled rapidly producing a fine, consistent grain structure. The total process time can be as low as ten minutes, removing the bottleneck of the BA process. The CA cycle, is specified in terms of five parameters: heating time, soaking time, cooling time, soaking temperature and quenching temperature. A typical time-temperature cycle is indicated in Figure 2.3. As with the BA route, there are a few disadvantages. Firstly, the process is less versatile, in particular no large steps in thickness or width can be achieved between successive coils. Secondly, if the strip breaks, the downtime and subsequent interruption costs can be very high. Thirdly, the coil must be completely cleaned, usually by gas electrolysis, before entry into the CA line, to prevent contamination marking the strip, during downstream processing.

2.2.4 Secondary Reduction

All intended packaging gauge products, up until this point, have had a similar history, but now the product can follow one of two routes. Depending on the required end properties of the coil, the plate can be either single reduced (SR), or double reduced (DR).

Single reduction

A single reduced (SR) product will be taken from the fully annealed state and given a light 'temper roll', imparting approximately 1% reduction in thickness. This final surface finishing treatment, has a dual effect. Firstly, it involves sufficient strain to avoid discontinuous yielding, which can cause unsightly stretcher strain marks (also called Lüdersbands), without adversely effecting ductility. Secondly, it imparts the final required surface finish, whilst improving strip shape. The temper mill usually consists of two, four-high stands and is of a similar configuration to the cold mill. However, the finish on the work rolls is of paramount importance. The first set of work rolls are either 'shotblast' for a non-directional finish or 'ground' for a directional surface finish. The second sets of work rolls are smoother, either being ground for a stone finish, or polished for a bright finish. A typical SR product, as described above, has a maximum tensile strength of 500 MPa.

Double reduction

The 1960's saw many technological and product developments, one of which was a double reduced (DR) product, section **2.1.1**. The technique of secondary reduction takes an annealed strip and imparts an 18 - 35% cold reduction, causing the tensile strength to increase to anything up to 650 MPa. This stronger material allows the use of thinner gauges, between 0.10 - 0.20mm. The forming characteristics of the strip are not greatly affected, although a slight decrease in ductility is usually noted. There must also be an element of directionality due to the low temperatures of the secondary cold reduction process.

2.2.5 Substrate Coatings

Any tin-mill product can be coated in two ways; the traditional electrolytic tinning (ETL), or electro-chromium coating (ECCS). Alternatively, the coil can be left uncoated, termed 'blackplate'.

Electrolytic tinning

The electrolytic tinning line (ETL) is essentially a continuous process where coils are welded head to tail, cleaned, pickled and passed through a plating section before being flow-brightened (if necessary), oiled and inspected prior to recoiling. The efficiency of the plating process is dependent on the cleanliness of the coil. To ensure the removal of rolling oils and other contaminates, the cleaning solution is a mixture of proprietary agents (usually polyphosphates), sodium hydroxide and wetting agents. Electrolysis (incorporating steel electrodes) is used to aid the process, allowing the strip to be either anodically or cathodically charged.

The pickling section is employed to remove the other contaminant oxides. Using a sulphuric acid bath at ambient temperatures, electrolytic pickling is most effective at removing the oxides and lightly etching the steel strip. The plating section is the most important area of the coating process, since the amount of tin deposited governs the corrosion properties of the coil. Corus Packaging Plus use a 'Ferrostan' process. The line consists of a series of vertical adjacent tanks, allowing the strip to pass consecutively through each one, increasing the coating thickness at each station. Within the tank, the anodes are of a minimum purity of 99.85% tin, the electrolyte consists of a stannous sulphate, phenolsulphonic acid and an organic addition agent. The sulphate provides the initial source of tin ions, the acid increases the solution conductivity and the organic agent facilitates the deposition of a coherent tin layer, which can subsequently be flow brightened. The electrolyte solution is circulated around the tank to maintain the plating consistency over time. The plate can be produced with differing coating weights on opposite sides, called a differential coated electrolytic tinplate, and as such it must be clearly marked to allow subsequent identification. Typically, a sodium dichromate solution is used to print a pattern of symbols, which can be decoded at a later date.

The tin coating after exit from the ETL has a dull matt finish. If the end product requires a shinier bright finish, then flow brightening or melting must occur. This process also causes a tin-iron alloy to be produced, which increases corrosion resistance. The strip is heated to a temperature just above the melting point of tin, then rapidly cooled before the coating touches a work roll. There are two methods of flow brightening. The first, resistance melting, uses a low frequency alternating current to facilitate the heating of the strip. The second, induction heating, uses a high frequency induction coil to rapidly heat the coil, by inducing eddy currents in the steel substrate. Both methods then rapidly solidify the tin coating, leaving a lustrous finish.

The oxide film on the tin coating is prone to discoloration, especially in humid warehouse conditions. This 'yellow stain' phenomenon, besides being aesthetically unappealing, hinders lacquer adhesion. To overcome these problems, a passivation treatment is required, depending on customer requirements and intended final product usage. It should be noted that some DWI grade steels require no such treatments, simply bypassing this section of the plant.

Electro chromium coatings

The second major coating, is an electro-chromium coated steel (ECCS). This coating is similar to the ETL in many respects, in that it requires cleaning, pickling, plating, oiling and recoiling; the differences lie in the bath compositions, the electro-depositing efficiency and the passivation treatments.

The cleaning and pickling processes are very similar to those previously mentioned, except that no current is applied at this stage, avoiding sulphate staining. The plating section, is where the main difference lies; the strip is passed through between two and five tanks containing an electrolytic solution of Cr^{6+} ions, forming a duplex metallic oxide/chromium hydroxide coating. The amount of coating deposited is lower than for the ETL, but the overall efficiency of the deposition is much lower, possibly due to excessive hydrogen evolution. The ECCS process does not require any extra passivation treatments, with consequent savings on further processing costs. At the exit of the line a thin film of oil is applied to allow movement of sheets at the

customers plant. These are either dioxysebacate oil (DOS) or butylsterate oil (BSO), although a third, acetyl tributylsterate (ATBC) has occasionally been used.

2.2.6 Polymer Coated Material

Corus Packaging Plus have two generic polymer coated products named Protact and Ferrolite. These are value added products, which consists of a substrate coated with a polymer film, generating a coated product which has a clean appearance and good corrosion properties, while still maintaining steel's good formability characteristics. The predominant UK production method is for the product called Ferrolite and its details are discussed below. The other product, Protact, is manufactured in a slightly different way, which is describe more fully elsewhere.[37]

Ferrolite

The Ferrolite line at Trostre was commissioned in 1992, and is shown schematically in Figure 2.4. The process coats a steel substrate with a polymer coating. The steel substrate is usually an ECCS grade; tinplate can be used for specific applications, although the tin coating may remelt under certain conditions. More recently, trials have being undertaken using pre-treated blackplate coils but the success of these experiments has yet to be determined. The polymer coating can be applied to both sides and is usually polyethylene terephthalate (PET) or polypropylene (PP). PP with a nylon top coat (PPN) has occasionally been trialled, but not commercially used; the polymer coating is generally in the 15-40μm range, although up to 200μm thickness is possible. The polymers used are either translucent or white, although other pigmented shades including gold, black and blue are being investigated. The applied films consist of a bonding layer, 5-10% of the thickness, whilst the remainder is bulk resin topcoat.[32]

Film application process

The coils are welded together on entry into the unit and then pass through an accumulator. Upon exiting the accumulator the substrate is scanned for welds and pinholes. The strip then enters the laminating section of the line where it is preheated by radio frequency induction. Electrostatically cleaned polymer film is then applied to

either side of the substrate simultaneously at the nip rolls. Monitors are undertaken to ensure the film is central to the coil and that the nip roll pressures are constant across the strip. The composite strip then enters a second induction heater, which melts the polymer film. It is then held at a temperature above the recrystallisation point of the polymer in a soak zone; this forms a strong intimate substrate-polymer bond. Extra heating may be required to obtain the necessary temperature regime, or conversely cooling can be achieved using air fans. Once the desired temperature for recrystallisation is acquired, the strip is rapidly water cooled, to quench-in the minimum crystallinity structure. A combination of air knives and squeegee rolls then dry the strip, before the edges are trimmed to ensure the polymer coating extends to the periphery of the strip. This laminating method produces a near fully amorphous structure in the film coatings, which has very low crystallinity. This ensures the polymer layer of the composite is formable and has good barrier properties in a drawn component. Upon leaving the laminating section of the line the material passes through an inspection station, where surface quality is audited. During the exit stage of the line, any defects or welds are Laser marked and removed before final recoiling and shipping to the end user.

Competitive laminating methods

The Ferrolite method of manufacturing film laminates is not the only system in existence. Toyo Kohan Co. Ltd (of Japan) also manufacture a steel-polymer laminate for beverage applications, with the most successful product (manufactured by Toyo Seikan) being the ultimate can (TULC). The Toyo Kohan method varies from the Ferrolite production process in many ways. Firstly, the polymer film is different in that no bonding layer is used and the bulk polymer is of a slightly different composition.[30] This film is a copolymer of PET and up to 18% Polyethylene isophthalate (PEI). Secondly, it is biaxially oriented, reportedly giving better strength and impact resistance.[38] The PET also has low permeability increasing barrier properties. The film application process is also different from the Ferrolite system discussed above. In order to achieve the necessary adhesion without the use of a bonding layer, the substrate must be heated to above the polymer's melting point and then rapidly cooled upon exit from the nip rolls. The resultant microstructure consists of an amorphous PET layer only in the vicinity of the substrate interface, whilst the

top layer remains oriented.[39] This amorphous layer is between 30 and 70% of the total film thickness depending on the required application and film dimensions. The main disadvantage of this laminating system is that it only uses one polymer-PET and the process will not easily allow dissimilar materials to be applied to opposite sides.

Rasselstein use a variation of the Toyo Kohan laminating method described above, named Andrafol and has the same laminating limitations. However, they also the facility to use a process route analogous to Ferrolite production, by incorporating a bonding layer into polymer films, thus allowing production of dissimilar polymers onto the same steel substrate.[40]

2.3 Can Making Methods

The major can making methods use either two or three piece production technology. Introduced earlier, each of these processes are discussed more fully below.

2.3.1 Three Piece Can Making

Three-piece cans consist, as the name suggests, of three pieces; a cylindrical body and two ends (the 'makers end' and the 'fillers end'). The makers end is attached to the body during manufacture, whilst the other is dispatched with the rest of the can, for use after filling. The body is made from a rectangular blank, which is welded to make the cylindrical body. Using a rectangular blank has many advantages. Firstly, the amount of waste produced is very low, efficiently using the blank or substrate. Secondly, since the blank is flat prior to joining, any decoration or printing is easily applied to this level surface, allowing superior appearance and finish. There are many slight variations and resultant effects to this process, including preheating, to prevent longitudinal bow. Morgan[10] discusses these variations in some depth. The final operation, once the cylinder has been produced, is to apply a flange to either end, easing the end attachment process. The end and body are then joined together in a double seaming technique. Finally the can is tested (under pressure) for micro-leakage, before being distributed to the fillers. Since the present project is primarily concerned with the forming of two piece cans, the joining of rectangular blanks in the three piece process, shall not be discussed further.

End manufacture

A round blank is stamped from a lacquered, and if necessary, printed metal sheet to produce the required size ends. Unlike a rectangular blank, the circular blank will always, by nature, have waste. A scrolled sheet produces minimum waste, and is favoured by most manufacturers. Scrap generation is greatest when each circle is enclosed within a square and at a minimum when the circles are in a close packed array. Once the round blank has been produced, it must then be contoured. This helps to strengthen and support the finished can, and also allows a lip or flange to be produced at the edge, to facilitate the joining to the can body. The 'traditional' end to

many food cans requires a can-opener, to remove the lid. Relatively recently, an easy open end (EOE) has been developed, allowing the whole lid to be removed by pulling a ring; this innovation has since been extensively improved and full aperture easy open ends are now commonplace for many cook-in sauces and semi-solid food ranges.

2.3.2 Two Piece Can Making

As previously mentioned, there are two major sub divisions of two-piece can making, although a third more recent development is also discussed. Respectively, these are drawn and wall ironed (DWI) and drawn and redrawn (DRD) with the last being draw stretch redraw (DSRD). Each was introduced in section **2.1.1**, but is now described in more detail below.

DWI: draw and wall ironed

Reynolds Metal Co. developed a forming process, called DWI, draw and wall ironed. This process allowed seamless two piece cans to be produced. A circular blank, initially aluminium but now predominantly steel, is drawn into a shallow cup, then redrawn to the required diameter, without affecting the wall thickness. After redrawing, the cup is pushed through a set of three ironing dies. Each successive die is narrower than the last, causing the wall diameter to decrease, typically from 0.24mm to between 0.08 and 0.15mm (for a 33cl can), and this being proportional to the resultant increase in the wall height. Schematic diagrams of the redraw tooling and ironing dies are shown in Figure 2.5. The process has a few drawbacks. Firstly, it requires copious amounts of lubrication,[10] and as such is providing environmental concerns about the collection and separation of the oil-water emulsion. Secondly, a DWI can requires thorough washing and organic protection (lacquering) prior to any contact with its contents, increasing the production cost per unit.

DRD: draw redraw

In this process, the blank is drawn into a cup, which is subsequently redrawn into one of a smaller diameter, and therefore greater height, indicated in Figures 2.6. The

greatest can height achievable in each drawing operation is governed by the LD_{rat} (limiting draw ratio), a ratio of cup height to diameter. It is affected by many factors both mechanic and material, which will be discussed more fully in section **5.2.3**. In the DWI process, the internal diameter of the can body remains constant, whilst the wall thickness decreases with increasing height. In the DRD process, the wall thickness remains uniform at each redraw stage. It can be seen that the DWI process offers material savings over the DRD process, due to wall ironing. However, the DRD system requires least lubricant.

DSRD: draw stretch redraw

The DSRD system is a redraw technique with a small amount of wall thinning midway through the forming procedure. The DSRD process uses sharper die and blankholder radii and also accommodates a larger die gap than the DRD and DWI systems. This process offers material savings over a conventional DRD procedure, the wall thinning reduces the material requirements by 18-20%,[41] and thus induces savings in the overall can costs. Additionally, the DSRD process does not require quite so much lubricant as the DWI method. The lubricant is usually removed by stoving prior to printing, thus alleviating some environmental concerns of the water and resources wastage. The DSRD process was refined in a joint venture between BST and Redicon, to produce a patented stretch redraw process. This system is being used to help manufacture the new RBS film laminated steel can project, which has recently been reviewed.[22]

2.4 Mechanics of Forming

The production of a cup from a flat disc is an inherent part of every two-piece can making method. It is a relatively simple process but its success is necessary before any further drawing or ironing can take place. The formation of this first cup is influenced by many factors; these can be split into two groups, material and mechanical issues.

2.4.1 Material Properties

There are many material properties which give an indication of the formability of that substrate, including yield stress, ultimate tensile strength and percentage elongation at failure, all of which can be derived from the resultant curve of a simple tensile test, as shown in Figure 2.7. A typical single reduced product would have a UTS of between 270 and 520 MPa and have elongations of up to 30%. The double reduced products has a 0.2% proof stress of 500-620MPa and an elongation of less than 2% However, for a more accurate evaluation of the formability we must consider one or more of the following:

Plastic strain ratio

One possible cause of failure of steel during a deep drawing operation is the onset of plastic instability (necking). A factor which controls the beginning of this necking phase is the r-value, or plastic strain ratio, defined as the ratio of width to thickness true strain, measured in tension. w_f and w_i refer to the final and initial sample width respectively, whilst t_f and t_i correspond to final and initial sample thickness.

$$r = \frac{\varepsilon_w}{\varepsilon_t} = \frac{\ln\left(\frac{w_f}{w_i}\right)}{\ln\left(\frac{t_f}{t_i}\right)} \qquad \textbf{Equation 1}$$

The measurement of thickness in a thin steel sheet is subject to considerable error. Therefore, if we assume constant volume (Poisson's ratio $\nu = 0.5$), then Equation 1 is re-written as:

$$r = \frac{\ln\left(\frac{w_i}{w_f}\right)}{\ln\left(\frac{l_f . w_f}{l_i . w_i}\right)} \qquad \textbf{Equation 2}$$

This r-value can be measured anywhere between the end of discontinuous yielding and the maximum load, see Figure 2.7.

Average plastic strain ratio

The plastic strain ratios or r-values are conventionally reported as an average plastic strain ratio, r_m (sometimes referred to as $\bar{r}$). This r_m value is an average of all the r-vales at 45° increments to the rolling direction.

$$r_m = \frac{r_0 + r_{45} + r_{90} + r_{135} + r_{180} + r_{225} + r_{270} + r_{315}}{8} \qquad \textbf{Equation 3}$$

Which simplifies to:

$$r_m = \frac{r_0 + 2r_{45} + r_{90}}{4} \qquad \textbf{Equation 4}$$

Planar anisotropy

The variation in r-value with rolling direction allows evaluation of the planar anisotropy along the sheet, indicated by the symbol Δr.

$$\Delta r = \frac{r_0 - 2r_{45} + r_{90}}{2} \qquad \textbf{Equation 5}$$

It should be noted that in a fully isotropic material, by definition r =1 (hence Δr = 0). For complex deep drawing operations a high r-value is preferred. A typical tinplate aluminium-killed steel grade has a r_m of between 1.0 and 1.4.

It should be noted that in double reduced (DR) materials no discontinuous yielding occurs and total elongation is low (typically less than 5%). This makes meaningful calculation of r-value, and hence r_m and Δr, very difficult.

Earing performance

The use of a cupping test, which forms a shallow cup in a simple drawing process can be used to measure the variety and magnitude of any earing that may occur. For the single reduced (SR) product three distinct ear types are observable. The first occurs below an optimum percentage thickness reduction, with the result being four ears at 0° and 90° to the rolling direction (R.D.) Around the optimum percentage reduction the ears are at a minimum, with six ears at 60° increments to the R.D. This minimum earing quality is very dependant on steel chemistry and applied total percentage reduction from hot rolled band gauge. For a 3165 steel grade the optimum percentage reduction to achieve minimal earing is reportedly 87%, although other factors such steel chemistry are known to affect the location of this point.[42] The final earing type occurs at percentage reductions, which are above the optimum value, here four ears are observable at 45° to the R.D. As logic would suggest, the further away the applied percentage reduction is from this optimum value, the greater the height of the resultant ears.

The other type of product available after cold reduction is the double reduced (DR) product. This product by nature always has very high percentage reductions applied, typically >90%. This almost exclusively limits any cupping test results away from the minimum earing levels, observable as four ears each at 45° to the rolling direction.

Work hardening exponent

Another measurable mechanical property is the work hardening exponent, n. The Lüdwick-Holloman equation (below) is only valid in the true stress, true strain region of the curve.[43] In this equation, σ, ε and k represent stress, strain and a constant, respectively.

$$\sigma = k\varepsilon^n \qquad \textbf{Equation 6}$$

$$n = \frac{\log \sigma}{\log \varepsilon}$$ **Equation 7**

Strain rate sensitivity

The Lüdwick-Holloman equation only predicts the work hardening exponent, n-value at one strain rate (i.e. speed of formation). Since all ferrous metals behave differently under different strain rates, a strain rate sensitivity value, m was developed.[44] This considers strain rate at two different positions, in combination with the stress at those points and is used to determine m. A typical m-value for aluminium killed DWI grade steels is in the region of 0.02.

$$\sigma = k' \left(\dot{\varepsilon}\right)^m$$ **Equation 8**

$$m = \frac{\ln\left(\frac{\sigma_2}{\sigma_1}\right)}{\ln\left(\frac{\dot{\varepsilon}_2}{\dot{\varepsilon}_1}\right)}$$ **Equation 9**

2.4.2 Polymer Coatings

The polymer coating for Ferrolite is usually polyethylene terephthalate (PET) or polypropylene (PP). A nylon coating has also been used in conjunction with PP but without much commercial success and shall not be discussed further.

Polyethylene terephthalate

PET is a semi-crystalline plastic of the polyester group and usually made by transesterification, the reaction between a terminal alcohol group and any ester group.[45] Pure PET, under ambient conditions, has a glass transition temperature (T_g) of 342 Kelvin and a melting point (T_m) of 529 Kelvin.[46] The PET repeat unit has a chemical formula of $[CH_2\text{-}CH_2\text{-}O\text{-}CO\text{-}C_6H_4\text{-}CO\text{-}O]_n$, which is indicated in Figure 2.8. PET exhibits a high tensile modulus and yet is still flexible enough to be formed into

deep-drawn shapes. The resistance to a variety of food and beverage products is reportedly very good, as is the polymer's resistance to chemical attack.[47]

The bulk polymer used for film laminate applications is essentially the standard PET homopolymer. A copolymer of PET and PEI (polyethylene isophthalate) is used for the bonding layer. The addition of PEI into the repeating unit causes a decrease in the melting point of the polymer, with further additions showing a proportional decrease in T_m. Some work has been conducted investigating the suitability of PEI as a copolymer in the bulk layer of the film. The PET/PEI copolymer has many advantages over the standard PET homopolymer. Firstly, lower stresses are induced in the coating during forming, allowing better adhesion and barrier properties. Secondly, the addition of PEI reduces the time to half-crystallinity of the copolymer, resulting in larger production windows during the laminating process.[47]

Polypropylene

The other regular choice of polymer for the coating of steel laminates is polypropylene (PP) which has a chemical formula of $[CH_2\text{-}CH\text{-}CH_3]_n$, with a typical arrangement shown in Figure 2.9. It reportedly offers high resistance to chemical attack and has beneficial processing properties due to its high percentage elongation at failure. However, PP's inert nature can cause substrate adhesion problems,[48] which can lead to problems during processing. The physical properties of PP vary with the tacticity and crystallinity of the polymer, typically the T_g is approximately 260K, whilst the T_m is about 460K. PP is an ideal polymer coating for harsh operating conditions, such as internal aerosol coatings. To assist with these difficult conditions the laminating process is capable of using up to 200 μm thick PP films. As with most polymers, the side chains can be arranged in three different ways, they may either be randomly ordered (atactic) or symmetrically along one side (isotactic) or in a regular alternating pattern (syndiotactic), this steric order greatly effects the crystallinity and resistance to chemical attack of the polymer. The polymer used for the Ferrolite coating has low atacticity, further improving PP's barrier properties.

Changes in polymer structure during heating

The changes that take place in a polymer during heating can be considered in many ways, one of which is the amount of molecular motion. Amorphous polymers are glassy solids at low temperatures because molecular motion is severely restricted. The temperature at which a polymer softens (when the free volume increases suddenly to a point where long range segmental motion becomes feasible) is known as the glass transition temperature (T_g). Immediately above this point, the physical properties are similar to rubber, where large elastic elongation can be accommodated by linear or lightly cross-linked systems. As temperature increases further, all types of molecular chain segmentation become possible; the molecules then assume a state of continuous random motion, which defines the 'melt state' of glassy polymers.[49] These changes can clearly be seen in the differential scanning calorimetry (DSC) trace indicated in Figure 2.10. This typical DSC trace for an amorphous PET film shows three main deviations from the underlying, but increasing, slope. These changes occur when the sample undergoes a physical or chemical change, detected by the resultant change in enthalpy. The first is the glass transition temperature (PET~75°C), which is an endothermic change. Energy is required to soften the polymer, allowing a relaxation of any stresses (residual or external) and long range chain movement. The second enthalpy change occurs over a temperature range of approximately 20°C, and has a maximum rate at 145°C. This change in structure is called the crystallisation point. Alignment of the polymer chains in the form of spherulites cause a lower energy, stable state, to be achieved. This process releases heat and causes a dip in the DSC trace. The resultant structure of the polymer is spherulitic lamellae, with long chains randomly bridged across the crystalline blocks. The final change in enthalpy during heating occurs at approximately 250°C, which is the melting temperature of PET. Energy is required to produce molecular segmentation that in turn leads to random motion. If the melt is heated much further above this temperature thermal degradation of the polymer rapidly begins.

Ageing of polymers

When a polymer is rapidly cooled from above its recrystallisation range, as in the Ferrolite production process, a non-crystalline amorphous structure is produced. Amorphous solids are not in thermodynamic equilibrium.[50] The excess volume

trapped between molecules is referred to as free volume. A system that is not in thermodynamic equilibrium will attempt to lower its energy state over time, primarily by continued packing of the structure. The changes in the structure of a polymer and the effect on mechanical properties is known as free volume relaxation, enthalpy relaxation or physical ageing.[51] The concept of ageing can be explained in terms of free volume and mobility.

The free volume concept states that in every system of particles, molecules etc., the mobility, M, primarily depends on the degree of packing, or in other terms on the free volume v_f. Consider cooling a polymer from a temperature T_0 that is greater than the glass transition temperature (T_g) of that polymer to some temperature lower than the T_g. Above the T_g the polymer behaves as a rubber or a fluid. Therefore, even at very low strains the mobility is large and the free volume must also be large. Upon cooling, there will be some simultaneous decrease in v_f and M.[52] At the glass transition point the polymer's properties change from those of a rubber to those of a glass. Therefore, the free volume must decrease. The mobility also decreases, but does not reach a low enough value to restrict all chain movement. It is this movement which allows ageing to occur. The amount and rate of ageing is governed by the magnitude of the free volume.

Ageing occurs in all glassy materials and is of particular importance in amorphous polymers.[53] It can also occur in the amorphous region of semicrystalline polymers.[54] Ageing causes a gradual densification and stiffening of molecular chains and generally produces a stronger, more brittle material.[55] This process has been reported on both polymer types investigated within this programme.[56, 57]

When the material is reheated above the T_g, it readily reaches thermodynamic equilibrium. By definition, the history of the sample is 'forgotten'; any previous ageing that occurred below the T_g has been erased. Ageing is a thermoreversible process that can be reproduced an arbitrary number of times on the same sample.[52] Literature also states that the phenomenon of ageing occurs at different rates depending on the polymer thickness.[58] Although this reference considers the difference between polymers of 0.5 and 25μm, it must be assumed that there is a

difference in the ageing rate in 15 to 40μm ranges used in typical film laminate applications.

An introduction to crazing

Crazing is a mode of localised plastic deformation, which usually, but not exclusively, occurs in all thermoplastics when stressed below the glass transition temperature, T_g of that polymer.[49, 59] When a stress is applied to a plastic, such as polyethylene terephthalate, crazes may be observed before fracture. Crazes are like cracks in that they are wedge shaped and form perpendicular to the applied stress. However, they may be differentiated from cracks by the fact that they contain material that is stretched in a highly oriented manner, perpendicular to the plane of the craze, indicated in Figure 2.11. This highly stretched material is referred to as fibrils. Another distinction between crazes and cracks is that the former are able to bear a load.[60] There is considerable evidence to show that there is a close connection between crazing and critical crack formation. At certain stress levels, crazes will form and it is possible for cracks to then nucleate in a craze and propagate through the crazed matter. Therefore, crazes act as an indicator to failure at high stress levels. However, it does not follow that all crazed material will subsequently fail in this manner. Crazing and crack growth are two related but distinguishable mechanisms. The following description of craze propagation and growth only considers a sample under a uniaxial stress field. Therefore, the introduction of biaxial stresses to a sample, as in the experiments to be delineated later, complicates the interaction of these mechanisms.

Craze nucleation and growth is a complex subject with no established criteria dealing with amorphous polymers.[61, 62] However, there is recognition that crazing is inherently a cavitational process leading to the suggestion that craze nucleation must involve a dilatational stress component.[63] To illustrate craze nucleation, it is simplest to consider a sample with a defect or notch under a tensile stress condition. As the sample is strained, plastic deformation occurs on the surface of the defect, leading to the development of high hydrostatic stresses at the tip of the yield zone. As the dilatational stress increases with an increasing size of yield zone, a critical stress is achieved. At this point, it becomes possible for microvoids to nucleate under high

major stress conditions. These microvoids subsequently grow into voids, which interconnect with the remaining ligaments. Stabilisation can then occur due to strain hardening caused by molecular orientation.[64]

After initiation the craze tends to grow along the plane normal to the applied major tensile stress. A craze can grow in two different ways: by craze tip advancement, an expansion of the craze periphery generating more fibrils, or by craze width growth, normal separation of the two interfaces behind the craze tip.[65] The standard mechanism of craze tip advancement is generally agreed to be the Taylor meniscus instability process.[60,64] Narrow wedge-shaped zones of plastically deformed and strain softened material is formed ahead of the craze tip. The deformed polymer constitutes a fluid like layer, into which the craze tip meniscus propagates. In contrast, the undeformed polymer outside this zone acts as quasi-rigid plates that constrain the 'fluid'. As the craze tip advances, fibrils develop by deformation of the polymer webs between the two edges and convolution of the meniscus around the strain hardened regions.[65] This method produces a continuous void structure between the two interfaces, which is interspersed with fibrils at regular intervals. However, an important feature for maintaining craze growth is that the strain must remain localised at an advancing craze tip by orientation hardening of interconnecting fibrils. Otherwise, void formation simply promotes general yielding of the material with voids.

Environmental stress cracking

The presence of certain liquids can initiate crazing at stresses far lower than a polymer's typical stress limit.[60] This phenomenon is known as environmental stress cracking (ESC) and frequently poses unanticipated problems during the service life of many polymers. This mechanism is defined as any condition in which an external (or residual) stress is imposed on a specimen that is in contact with an external (usually liquid or vapour) environment; it is the combination of the stress and the liquid medium which gives rise to premature failure. This is often seen under conditions that would otherwise appear to offer little threat to the material if either the stress, or the environment were eliminated.[49] ESC is not a direct form of chemical attack and is not directly associated with chain scission. Its effects have been detrimental to the

performance of amorphous polymers stressed in the presence of organic solvents. The visible damage that occurs in ESC may vary considerably. In some cases a single defect, once initiated, can propagate rapidly through the sample; in other situations macroscopic crazing occurs, sometimes visible as whitening in the region of maximum stress. This has resulted in an additional term *environmental stress crazing.* However, these two terms should not be used interchangeably, purely on subjective evidence of damage zones in ESC failures.[49]

The theoretical mechanism of premature failure by environmental contact is based upon the assumption that the active medium penetrates any microscopic defects introduced by stress and diffuses into the material at the stress concentrator. This creates a greater stress concentration and accelerates the progression of the damaged zone. Failure will then occur if the localised load bearing characteristics of the crazed material becomes inadequately resistant to the increasing stress concentration. Environmental stress cracking is a well reported phenomenon regularly encountered during production and subsequent storage of PET bottles.[51] It has been found that the polymer when in contact with a sodium hydroxide based solution, such as those found in cleaning detergents, can rapidly fail through an ESC mechanism. This information shows that crazing of PET by either ESC or a standard craze initiation route is an inherent problem to which many applications of this polymer are susceptible. Crazing in polypropylene is less well reported. Generally, PP is much more resistant to environmental stress cracking than PET.[66] Additionally, ESC in PP is dependent on its physical properties. Craze susceptibility increases with tacticity and crystallinity.[67] Both of these factors are minimised in film laminated materials. This suggests that ESC would be less likely in a PP film laminated steel than one coated with PET.

The stress cracking and crazing of polymers in the presence of an active liquid environment is a well reported phenomenon.[68, 69, 70] Significant progress into predicting which organic liquids cause crazing with which polymeric coatings has been made in recent years.[71, 72] One of the most accurate methods suggested is to compare solubility parameters of the organic liquid and craze susceptible polymers.[73] This method considers the total solubility parameter (δ_t) of a material, which is

derived from three contributory factors; dispersion (δ_d), polar (δ_p) and hydrogen bonding (δ_h) solubility parameters. Their relationship is shown in Equation 10.

$$\delta_t^2 = \delta_d^2 + \delta_p^2 + \delta_h^2 \qquad \textbf{Equation 10}$$

If the total solubility parameter of the organic liquid and the investigated polymer are similar then environmental stress cracking is deemed likely. The further apart δ_t between the organic liquid and the polymer the less likely environmental stress cracking is to occur. This general relationship has proved robust for a number of liquid polymer environments.[74, 75]

2.4.3 Mechanical Forming Issues

Deformation issues markedly affect forming properties of deep drawn parts. A typical schematic diagram of tooling involved in formation of a first cup from a flat disc is shown in Figure 2.12. Each of the critical areas is discussed more fully below. All comparison of the following literature assumes all tooling is in perfect alignment and lubrication levels within experiments are consistent.

Disc size, commonly known as **cut edge**. The diameter of the disk governs the amount of material available for forming. Industrially, can makers want to minimise cut edge size for a given can height to enable more cans to be produced per unit area of material.

Punch diameter in combination with the cut edge dimensions governs the **draw ratio** of the cup produced. The draw ratio is calculated as shown in Equation 11. The maximum draw ratio for a given process is called the **limiting draw ratio** and is also shown in Equation 12.

$$D_{rat} = \frac{C_{ed}}{P_{dia}} \qquad \textbf{Equation 11}$$

$$LD_{rat} = \frac{C_{ed\ \max}}{P_{dia}} = \frac{C_{ed}}{P_{dia\ \min}}$$ **Equation 12**

Punch nose radius, the profile of the top of the punch in contact with the sample. Literature suggests that, for a drawn shell following the DRD process route, the punch nose radius should be at least 15% of the punch diameter.[76]

Punch Speed governs the strain rate at which the disc is deformed. Can makers like to keep this value high to increase throughput of their plant. Chung and Swift suggest higher punch speeds decrease the amount of failures caused by tear-off and therefore decreases machinery downtime.[77]

Die clearance is defined as the amount of room between the sample and the die when the punch travels through the tooling. It is usually given as a measurement or a percentage.

$$Clearance = \frac{\left(\frac{D_{dia} - P_{dia}}{2}\right) - T_i}{T_i} \text{x}\,100\%$$ **Equation 13**

If this value is negative, then ironing will occur during formation of the cup. Small values may not allow for any thickening of at the top wall. Very high values combined with a tight die radius can induce stretching of the cup wall. It has been suggested that clearance should approximate:

$$Clearance = T_i \sqrt{LD_{rat}}$$ **Equation 14**[76]

Die radius is the profile of the die, which is in contact with the sample during bending and unbending of the disc into the cup wall. The acceptable values for this variable shows large amounts of disagreement in the literature. For drawing of a lacquered steel or tinplate one paper suggested that the radius should be kept as high as possible to minimise bending losses.[76] Panknin also states that the radius should be

kept between 5 and 10 times the total material thickness, with an optimum multiple of eight. Other sources suggest radii as low as twice the material thickness can successfully be used for polymer film coated materials.[78] However, at low multiples of material thickness the deformation is likely to change from one of predominantly drawing to one incorporating a degree of stretch in the cup wall.

Blank holder force governs the amount of retardation exhibited by the sample between the die and blank holder during drawing of a cup. Insufficient blank holder force causes excessive wrinkling up the cup wall and may quickly damage tooling. Conversely, too high a blank holder force causes failure through tear-off. The range of blank holder force, between these two extremes, through which a cup can be successfully produced is known as the **operating window**. The above ranges of blank holder force and their effect on cup formation are shown in Figure 2.13. Most literature agrees that the blank holder pressure applied to a component should be just sufficient to remove trace wrinkling.[76,79] However, other blank holder forces within the operating window have been considered.[78,80] Gotoh et al states that a cup produced at higher blank holder force causes a thinning of the cup wall by a stretch draw process. This results in a deeper cup than those produced at a lower blank holder force.

There is little consistency in literature about quotation of blank holder retardation as a force or a pressure. The problem exists because as the cup is drawn a decreasing sample area is in contact with the blank holder, changing the applied force, in the case of a constant pressure gauge or vice-versa. Although conversion from force to pressure is not difficult, as long as cut edge and draw ratio are known, it is likely to magnify errors when comparing one paper with another.

Forming of laminates

The Ferrolite production process incorporates the use of a thin bonding layer to provide the required substrate-bulk polymer adhesion properties. This bonding layer is co-extruded simultaneously with the coating, and varies depending on with which bulk polymer it is to be applied. The bonding layer for the PET polymer is a copolymer of PET and PEI. The thickness of which can vary between 2 and 5 μm

depending on bulk polymer gauge. The PP bonding layer is an acid-modified PP polymer, which has a thickness of approximately 10% that of the bulk PP polymer. The considerations of substrate, polymer and bonding layer all have to be examined when examining the forming process. The effect of the polymer on the forming of these laminates is not fully understood. It is known that the polymer has a lubrication effect during the forming process, but the exact mechanisms have not been fully investigated. It is also thought that some drawing mechanisms cause a degree of crystallinity and orientation in the cup wall, resulting in varied barrier properties.

There is evidence to suggest that certain film laminated material exhibits stress induced crazing when dome deformation is applied.[81] This is seen as an elliptical whitened region on the internal clear coating of domed cans. It is also stated that the addition of a petrolatum wax to the surface of the laminate, prior to deformation, reduces the magnitude of the crazed region. However, the crazing phenomenon is exclusively located in the dome region and is not visible on the drawn can wall.

A number of papers published in Japan have considered adhesion measurement and control of steel polymer laminates.[82, 83] However, little attention is given to deformation of these materials. The remaining published information on deep drawing of polymer coated steels concentrates on the modelling of the DWI can manufacturing route.[84, 85] These studies provide useful background to forming of thin gauge steel polymer laminates, but do not consider redrawing or biaxially stressed deformation modes.

Other than that previously presented there has been little published information on the deformation of steel-polymer laminates, although some analysis has been carried out on steel-polymer-steel sandwich materials for use in the automotive industry.[86, 87, 88, 89] The results of these investigations are of little practical value to thin gauge can making applications, because the polymer is not in intimate contact with the die, the substrate gauges in question are larger and the polymer coating thickness is much lower as a percentage of the total laminate.

2.5 Conclusions

Drivers in can making

1. History has shown that most foodstuffs require packaging and the metal can is the most popular type. A combination of production costs, container durability, consumer requirements, weight and environmental considerations act as drivers to the canning industry.
2. For all major two-piece can making technologies a cup is required to be drawn from a flat disc. Many factors influence the dimensions of this cup but they can all be grouped into either material or mechanical issues.

Material issues

3. Film laminate materials for packaging applications consist of a substrate and one or more polymer coatings.
4. The substrate is usually a double reduced low-carbon steel with gauge range of 0.12 to 0.4mm and has an electro chromium coating (ECCS) to passivate the substrate surface. Tinplate based or treated blackplate substrates have been used but not to the same extent as ECCS substrates.
5. The polymer coating is typically polyethylene terephthalate (PET) or polypropylene (PP) and is usually clear or white pigmented. These polymers are 15 to 40μm in thickness, although up to 200μm is possible.
6. The polymer is generally applied in film form to the substrate, depending on which manufacturing methodology is being employed. Recent developments have seen direct extrusion of the polymer onto the substrate.
7. Measurement of substrate formability can be achieved through many parameters including r, n and m values. However, for double reduced steel grades the low elongation to failure makes accurate assessment of these values very difficult.
8. The change in formability of a material through the addition of a polymer to the substrate is not clearly understood. A change in frictional characteristics has been detected.
9. An amorphous, crystalisable polymer structure changes with time. This change is caused by excess free volume trapped in the polymer lattice and its subsequent

relaxation. This phenomenon is referred to as ageing and is shown to occur in PET. The rate of ageing changes with polymer thickness.

10. Heating of an amorphous polymer from low temperatures cause results in several changes in structure. These are referred to as the glass transition temperature, recrystallisation point and melting point.
11. The glass transition point is defined as the temperature at which the polymer softens and long range segmental motion becomes feasible. Heating of a sample to this temperature causes the polymer to 'forget' any previous thermal history and erase any ageing effects. Hence, it is a thermoreversible process and can be theoretically repeated an infinite number of times.
12. All thermoplastics exhibit localised plastic deformation when stressed below the glass transition point. This deformation manifests as crazes; wedged shaped cracks that contain highly stretched fibrils of material perpendicular to the plane of the craze.
13. Craze nucleation and growth has no established criteria dealing with amorphous polymers. It is inherently a cavitational process, which causes microviods to form. These then grow into voids and interconnect with the remaining ligaments.
14. Craze tip advancement is generally agreed to follow the Taylor meniscus instability process. Narrow wedged-shaped zones of plastically deformed and strain softened material occurs ahead of the craze. The craze tip then propagates into the craze tip meniscus.
15. The presence of certain liquids can initiate crazing at far lower levels than the polymers typical stress limit. This phenomenon is called environmental stress cracking and frequently poses problems during the service life of many polymers. The mechanism is defined as any condition in which an external (or residual stress) is imposed on a sample in contact with an external (usually liquid or vapour) environment.
16. There are many reported examples of environmental stress cracking occuring in PET material.

Mechanical issues

17. The most important mechanical issues are related to tooling. Parameters of interest are cut edge, punch diameter, punch nose radius, stroke speed, die clearance, die

radius and blank holder force. Of these, literature mostly concentrates on the latter two factors.

18. The ratio of die radius to material thickness is often quoted as an important factor. The optimum value is around eight. Although different sources suggest values between two and twenty.
19. It is generally agreed that blank holder force (or pressure) should be kept at a minimum value, which just eliminates wrinkling around the top wall of the cup. However, a range of blank holder values between wrinkling (too low) and tear-off (too high) have been investigated with a degree of cup wall stretch identified at higher forces.
20. Literature suggests the punch nose radius should be at least 15% of the punch diameter. Values lower than this are likely to cause wall thinning
21. There are conflicting accounts of the limiting draw ratio for tinplate and packaging gauge steels. Reported values were in the range 2.0 to 2.5.
22. No values of limiting draw ratio for film laminated steels were found to be reported.

3 Programme Objectives

It can be argued that the two most important procedures in the production of two piece containers are deep drawing of the cylinder and forming of the dome. A container needs to be drawn to the required height from a specified sample and has to stay within guidelines set for contamination of the container contents after filling and before consumption. The drawn cylinder must be sufficiently tall to meet the required height, but wastage due to trimming of over-engineered cans must be minimised. The doming operation adds strength to the base of the can to allow pressurisation. It is therefore essential that a successful dome be achieved for safety reasons.

It is the above two areas of the can making procedure that are to be focussed upon in this research. The first section; drawing of the container is simplified to the first cupping stage from a flat disc, to allow direct comparison of one material with another. The second section is to concentrate on the doming operation and any associated polymer coating integrity issues.

Considering the above and conclusions reached in the literature survey, the programme objectives are as follows:

A1. To consider how a range of film laminate specifications behave under a simple drawing operation and to relate the drawn cup height to the variables under analysis. The four main groupings, which are expected to influence forming characteristics are:

- Substrate properties (gauge, strength, passivation)
- Polymer coating (type, thickness, colour)
- Lubrication (type, quantity)
- Tooling parameters (sample dimensions, punch and die specifications).

A2. Once the information gained from Part A1 has been understood and quantified it is then anticipated a predictive equation for drawn cup height could be developed, for a range of film laminated materials.

B1. The second area of research is to focus on the doming and coating integrity issues highlighted in the literature, concentrating on the stress whitening visible around the dome of certain film laminate materials. The following areas allow a systematic understanding of the problem:

- Develop a method of reproducing the crazing in the dome of film laminated materials
- Suggest a mechanism for appearance of this phenomenon
- Understand the influence of lubrication on craze development
- Investigate the effect of prestoving of samples, prior to forming, on craze development
- Highlight ways of elimination or prevention of the problem.

4 Materials and Experimentation

Materials used throughout this doctoral programme were multi-layered composites. These composites usually consist of one or more of a combination of substrate, passivation treatment and polymeric coating. The substrate can be of varying thickness, strength or temper. The passivation treatment can be tinplate, electro-chromium coated (ECCS) or the substrate could simply be left clean (blackplate). The polymer coating can be one of many different types, thicknesses and colours. Typically, polypropylene (PP) and polyethylene terephthalate (PET) films are used, with a thickness range of 15 to 200μm. Commercially, the film is either transparent (referred to as clear) or white pigmented. For ease of reference, materials used within this document have been split into the two main experimental sections; drawing and biaxial stressing of film laminated materials.

4.1 Drawing of film laminated materials

4.1.1 Materials for investigation

All materials under investigation, within this section are shown in Table 4.1. The plethora of film laminates considered all have uses in isolating specific factors that influence their forming characteristics. (Full experimental details are shown subsequently in section **4.1.5**). Laminate no. 1 was used for development of the standard operating procedure and for assessment of scatter and reproducibility of results. Variables 2-4 and 5-7 were used to determine the influence of passivation and coating type on drawn cup height, for two different substrate gauges. The remaining variables were used to assess how either substrate gauge and strength or polymer properties influence formability of laminate materials.

Almost all materials were produced with standard preheat, postheat and soak temperatures on the laminating line. The only exception was material no. 16, which was produced at higher postheat and soak temperatures. This allowed comparison to material no. 14, which had identical specification, but was laminated with a standard (lower) temperature in the critical zones.

4.1.2 Sample preparation

Cutting up

Typically, laminate material is produced in a coiled form and as such has to be cut into a more manageable size for further evaluation. The coil was usually cut into sheets of approximately 900mm^2, depending on laminated coil width, on an industrial shear line. These sheets were then stacked on to a pallet and transported to the research facilities. For the cupping experiments these sheets were then cut into strips for sample blanking. The strips were always orientated across the rolling direction; this allowed several discs to be taken from different points across the coil width from one sample strip. Four disc diameter sizes were investigated, 135, 125, 120 or 100mm. For each of these, the strips were 155, 145, 135 and 125mm in width respectively.

Labelling

Once the required strip dimensions were achieved they were labelled with identifying marks prior to blanking of the discs. Samples were taken from six positions across the width, two each from the edge, edge-centre and centre positions of the strip. This is more clearly indicated in Figure 4.1. Samples were labelled with three numbers. The first was used as reference to the sample position within the original coil. The second indicated the intended cupping order. The third (if required) identified what blank holder force was to be used for production of that disc. This nomenclature is shown in Figure 4.2. All identification was located in the central 67mm of the disc. This ensured that the inks did not interfere with the draw of the cup side wall.

Blanking

Four diameter disc sizes were used 135, 125, 120 and 100mm. The first three sizes were blanked on a Bliss 10 blanking press with the appropriate tooling installed. The last were cut on a Carmilles CNC lathe to a specified and repeatable programme. Discs were checked for out of roundness or any other defects acquired during blanking and then randomised in preparation for cupping.

Lubrication

To facilitate the cupping procedure, a small amount of lubricant was added to each disc. Before application of the lubricant discs were weighed in packs of 10. For the majority of the experiments a DR9 oil and isopropyl alcohol emulsion was used as the lubricant; mixed in a ratio of 1:1. This was dropped onto the surface of each face of the disc with a pipette and spread onto the remaining surface area with a cloth, prior to reweighing of the sample groups. Comparison of each group of disc weights was then undertaken to ensure lubrication quantity was similar for all samples.

4.1.3 Forming procedure

The current standard operating procedure for the Roell & Korthaus cupping press is located in Appendix I. This document highlights the acceptable procedure for cups produced for quality control and customer complaint issues. However, there is no mention of lubrication or production procedure of large numbers of samples. Hence, using the current operating procedure is no guarantee of consistent or reproducible results.

Cup height measurement

Once the required cups have been produced they need to be measured to allow comparison with one set of experiments and another. To do this, the cups were randomised for a second time and then measured by UKAS accredited equipment for height and earing. For each cup, height measurements were recorded as average minimum (H_{min}), average maximum (H_{max}) and the difference between the two, ΔH (or earing). Division of ΔH by the minimum cup height gives a representation of percentage earing, see Equation 15.

$$\%\ earing = \frac{H_{max} - H_{min}}{H_{min}} x\,100\% = \frac{\Delta H}{H_{min}} x\,100\% \qquad \textbf{Equation 15}$$

The maximum value was calculated by identifying the four peaks of each ear on the cup and averaging these values. The average minimum was calculated for each trough

at the base of every ear. From a can makers point of view it is the minimum earing value that is of most importance as this indicates the shortest point of the cup and ultimately governs the amount of material wasted through trimming.

Tooling options

There are a number of different tooling sets complimentary to the Roell and Korthaus cupping press. The available die and punch dimensions are summarised in Tables 4.2 & 4.3. For the reproducibility of results analysis, which is described subsequently, tooling set A was used. For the majority of the cupping experiments all internal die diameters (D_{dia}) were identical at 67.488mm and only the die radius (D_{rad}) changed. The remainder of the tooling sets available were used for later work analysing the influence of draw ratio and punch diameter on cup dimensions.

4.1.4 Scatter and reproducibility of results

Before embarking on any meaningful research it was paramount to assess variability, and hence reproducibility, of results generated from the Roell and Korthaus cupping press. The first experiment looked at variation caused by machinery and the second related to material characteristics. Finally, the variation caused by lubrication was investigated.

Variability of machinery

To analyse any change in cup height with time 120 cups were produced continuously from ambient machinery oil temperature of approximately 16°C. The material chosen for this experimentation was a 0.17mm DR620 substrate laminated with identical 15μm PET coatings on both sides, material no. 1 in Table 4.1. The use of an identical polymer coating on both sides eliminates variation caused by film type, thickness or colour. All samples were taken from the centre of the coil to produce minimum amounts of variability in substrate strength or gauge. Tensile measurements taken at random points along the centre line are shown in Table 4.4. Samples were then randomised prior to lubrication and cupping. Cups were generated using a constant blank holder force of 19.6kN and the tooling set A in Table 4.2. The discs were put

into the machine at 30°, 90° or 150° relative to the die housing. This allowed further evaluation of any tooling misalignment and its effect on forming behaviour. Resultant cup heights from this experiment are shown against sample orientation (relative to the die housing), cupping order and machinery oil temperature in Figures 4.3, 4.4 and 4.5 respectively. Figure 4.3 shows how minimum cup height changes with sample orientation. There are no obvious trends, which suggests there is no misalignment between punch and die. If the punch wasn't concentric with the tooling lop sided cups would result, which would be easily visible during production and skew of the earing results. Figure 4.4 shows how minimum cup height varies with cupping number. Generally, the results show a scatter about a mean, with no definite trends detected. Figure 4.5 shows how minimum cup height changes with machinery oil temperature. This graph has similarities to the previous figure because the results are plotted in the same order. However, this time the x-axis does not increase linearly because of temperature losses through conduction and convection within the machinery. The variability observed of ±0.37mm, appears to be a consequence of the small differences in processing. The random nature of the scatter in data is confirmed by considering the histogram shown in Figure 4.6. The results clearly follow a normal distribution about a mean. The results represented in this diagram consider every ear on each cup, not just the average values as indicated previously.

Variability caused by material

To investigate effect of sample position across and along the strip two experiments were combined. Firstly, the above data was plotted to show how cup height changes with sample position along the strip, shown in Figure 4.7. (N.B. All data from this experiment was from discs taken from centre-only positions within the parent coil.) This shows excellent consistency in results between the centre pair of discs and minimal variation in cup height along the length of the investigated area, following a slight periodic sinusoidal pattern. Secondly, an experiment was devised to look at the effect of sample position across the strip on cup height. Samples were taken from six equal positions across the strip (refer to Figure 4.1); then marked, lubricated and randomised before cupping in a continuous run. Cup height is shown against sample position across the coil width in Figure 4.8. The standard deviation of average minimum cup height considering samples from all positions across the width is

0.178mm This represents a 0.02mm increase compared with the centre only samples, from the previous experiments.

This low amount of variation across the strip is not unsurprising. During Ferrolite production edge trimming is used to ensure the polymeric coating extends to the periphery of the coil. Edge rundown, which causes largest variations in substrate mechanical properties, is usually confined to the first few millimetres on each edge of the strip.[90] Therefore, the disc cupped from the edge position is usually sufficiently remote from the original coil edge to negate large substrate variability in film laminated feedstock.

Variation caused by lubrication

Once all differences caused by mechanical and material effects had been considered, operator error must then be assessed. An operator following the correct operating procedure can only realistically alter the outcome of the experiment in two ways; applying incorrect/inconsistent amount of lubricant or not correctly placing the sample in the machine. The latter of these becomes self evident by the resultant pinched or lop-sided cup. The former however is more difficult to quantify.

A set of experiments was conducted by applying known amounts of lubrication to four sets of discs before cupping. The four states investigated all used material no. 1 in Table 4.1 and were, no lubrication, 0.4 to 0.5mg per disc, 0.8 to 0.9mg per disc and flooding of the sample. The central two lubricant weights were applied by placing three or six drops from a pipette onto each side of the disc and then wiping on with a cloth. The final state was achieved by submersion of the disc in an oil/alcohol emulsion and then placement on a rack to allow excess to drip off. This method effectively resulted in a disc swamped with lubricant. For each data group the discs were weighed and an average taken for the set. This is shown in Table 4.5. The data sets are quoted in ascending quantity. The groups show average applied lubrication weights of 0, 0.44, 0.86 and 2.1mg respectively. The standard deviations were 0, 0.03, 0.07 and 0.17. Applying three drops to each side of the disc showed most consistent lubrication weight. The resultant cup heights for these four groups are shown in Figure 4.9. Problems were encountered with no lubrication, with more than 60% of

the cups failing to draw. For the remaining data sets, the immersion method showed least variation in cup height, but measurement of the samples proved difficult due to the coherent nature of the excess oil. The next best consistency in cup height was achieved with the three drops data set. Therefore, considering both consistency of application and drawn cup height variation, the three drops method was chosen as the standard application procedure. To ensure repeatability it was also decided to weigh discs in groups of ten in all experiments, before and after lubrication, to act as a safety mechanism when comparing results.

Revised operating procedure for Roell & Korthaus Cupping Press

All of the above results show how cup height is influenced by machinery, material and operator variability. Therefore a consistent repeatable operating procedure needs to exist to minimise variation in results and allow comparison with one set of results and another. This research highlighted considerable differences in current procedure and developed the following for use in all further experimentation for standard cupping on the Roell & Korthaus Press.

Sample preparation:

Samples must be marked to identify the original position of the disc within the original material, the cupping order and the required blank holder force. Discs must be weighed in groups of ten before and after lubrication to ensure consistent amounts of oil emulsion are applied to each disc. Acceptable average lubrication weight per 10 discs is 4 to 6 mg. Samples must then be ordered in preparation for cupping.

Machinery settings:

- ◊ Normal redraw tooling
- ◊ High speed on
- ◊ Nitrogen bag accelerator open
- ◊ Manual speed settings at zero
- ◊ Tooling size must exceed total laminate thickness.

Experimental procedure:

1. Place sample in locator ring, rolling direction away from the operator (i.e. towards the back of the machine).
2. Lower die and it's housing, locking handle into place by pushing away from operator. N.B. Machinery will not operate if micro-switch is not contacted.
3. Press both start buttons.
4. Set blankholder pressure to desired level, by adjusting the 'clamping pressure' dial.
5. Slowly open manual speed setting to 9 or 10 on the dial.
6. Wait for dial on top of the side box to reach 45.
7. Press green start button **once**.
8. Depress the 'cup extraktor' button until an audible click is heard.
9. Remove emerging sample from the top of the die housing.
10. Unlock the locating lever by pulling towards operator, lift the housing up and secure with clamp.
11. Repeat from 1. For each separate cupping test

Assessment of reproducibility

To assess the repeatability of this operating procedure a set of experiments was duplicated on separate days under identical conditions. On each occasion 50 cups were produced from material no. 12 in Table 4.1. This ensured that results were repeatable for more than one, and hence all, film laminated material. Minimum cup height for each repetition is shown as a frequency histogram in Figure 4.10. The standard deviations of the minimum cup height for the first and second experiments were 0.19 and 0.15mm respectively. Average minimum cup heights were 38.19 and 38.21mm. These experiments show good repeatability and reproducibility for the Roell and Korthaus cupping press when following the previously highlighted standard operating procedure.

4.1.5 Experimental investigation

In order to examine fully critical factors influencing drawing of film laminated materials it was necessary to isolate areas of interest. In an attempt to facilitate this approach, experimental investigation was split into three main groups; effect of substrate properties, effect of polymer coating and effect of tooling parameters. The

first two considered material specifications by individually examining the influence of substrate and film properties and how they changed forming characteristics. The last group isolated mechanical effects exclusively associated with tooling specifications and sample geometry. A final section, effect of lubrication examined more closely how lubrication weight and type altered drawn cup dimensions. The following introduces the experimental approach for each of these sections.

Effect of substrate properties

Three factors were investigated within this section, substrate coating/passivation, substrate thickness and substrate strength. The first set of experiments compared three different coating states based on the same substrate, uncoated material, lacquered ECCS and a standard film laminate specification. Minimum cup heights were established for two substrates on 0.195 and 0.26mm thickness, with nominal strength of 620MPa. Comparison of failure rates, as a total percentage of original discs and frictional characteristics were also conducted. The second set of data assessed cup height of a range of substrate thicknesses all laminated with the same polymer films and based on a substrate of nominal strength of 620MPa. The final experiments in this section assessed cup height of a range of film laminate materials of differing substrate strength with constant thickness of 0.19mm

Effect of polymer coating

The majority of film laminated materials are either polypropylene (PP) or polyethylene terephthalate (PET) based and are in the thickness range of 15 to 40μm. The coating colour can be either clear or white pigmented. Additionally, the polymer film can be laminated over a range of temperatures in the production cycle. Four types of experiments have been designed to look at each of these areas. The initial set of data compared a standard PET and PP film laminate material to detect any differences in forming characteristics. The second experiment considered PET and PP laminated materials with different coating thickness within the range 15 to 40μm. The third experiment compared white and clear coatings on opposing sides of the same laminate and how orientation of the sample relative to the die radius effects drawn cup height. Complimentary analysis of surface topography and composition will also take place to assess how TiO_2 particles interact with the tooling during forming. The final set of

experiments will consider drawn cup height of two PP laminates produced at two different commercial temperature regimes, which represent typical PET and PP film laminate production routes.

Effect of tooling parameters

Mechanical issues influencing forming are all grouped into this subsection. Four areas were chosen for assessment, blank holder force, die radius, cut edge and punch diameter. The range of blank holder force through which a cup can be successfully produced is termed the operating window. This window was calculated for a broad spectrum of laminate specifications and cup heights at various points within the range is to be analysed. For one specific laminate material based on a 0.18mm DR580 substrate a range of cups were produced at different cut edge dimensions, external punch diameters, punch nose radii and die radii. Results from these experiments were then be combined to assess how factors such as draw ratio and tooling specifications alter drawn cup height.

Effect of lubrication

The final experimental work in this section considers lubrication. This was to be achieved in two ways. Firstly, by considering how varying lubrication weight affects cup height for the same specification material. Secondly, a range of commercially available lubricants used within the packaging industry was applied to sets of material in even quantities and drawn minimum cup heights were assessed. Comparison between the various lubricants and their influence on deep drawing was then conducted.

4.2 Biaxial Stressing of film laminated materials

4.2.1 Materials for investigation

For the majority of the experimentation conducted in this section a 0.17mm ECCS DR620CA substrate, laminated on both sides with 15μm clear PET was analysed. This was produced under standard film laminating conditions (section **4.1.1**) for a PET polymer on an ECCS passivated substrate. A summary of material details is shown as no. 1 in Table 4.6. The second material considered in this section (no. 2 in Table 4.6) has identical specifications to the first, but was tested hours after production and not months as in the previous case. There was a small amount of ancillary testing undertaken on a blackplate based laminate (no 3 in Table 4.6). This had the same nominal specification substrate as the previous material, but bypassed the ECCS coating line. A 25 μm white PET and 20 μm clear PET coating was then laminated to either side of the substrate.

4.2.2 Sample preparation

Identical cutting up and blanking procedures were followed as in section **4.2.1**. However, labelling and lubrication methodology differed. In a doming operation (or simulation), which has a biaxial stress applied, the area of interest is in the central region of the sample. Therefore, all identifying marks must be on the periphery of the specimen, as shown in Figure 4.11. This has a dual purpose; firstly, it allows analysis of the deformed region without impairment of the view by the identifying marks and secondly ensured no ink-polymer interactions influence the results. Samples were labelled with information to record the age of the laminate plate, the temperature of prestoving (if applicable), the time period of prestoving, the type of lubricant applied and the degree of deformation.

Lubrication issues

Lubrication is complex issue when biaxial stresses are applied to a film laminate material. This is compared to the previous section where its presence simply facilitates the success of a drawing operation. With biaxial stressing of film laminates and any subsequent polymer whitening during deformation, two distinct lubrication

issues are highlighted. The first considers lubricants applied to the substrate after passivation treatment, which then became trapped between the layers of the laminate. This is shown more clearly in Figure 4.13. The second issue is the application of lubrication to the surface of a laminate prior to deformation. Experiments designed around these lubrication variants are shown more fully in section **4.2.5**.

4.2.3 Development of a simulation method

Once it became clear that the issue of crazing on can domes was of significance, a method to simulate the problem needed to be developed. It was also apparent that the production of numerous actual cans to analyse the phenomenon would be costly and time consuming. Therefore, a quicker and far simpler method of craze reproduction was needed to fully understand the factors affecting the crazing of film laminated materials. Three methods of craze simulation were investigated, all using a known 'problem' plate, material no. 1 in Table 4.6.

Detection of stress whitening

The first type of test to be investigated was a simple tensile load applied until material failure. This test was carried out on the Instron 1162 at Materials Engineering Dept, University of Wales Swansea. Samples had approximate dimensions of 150mm by 30mm. These were divided into three groups; the first was used as a control, the second group had a thin film of Dioxylsebacate oil (DOS) applied to the sample surfaces and the third had a similar amount of petrolatum type wax added. The groups were then sub-divided into thirds and the tensile test applied at three different speeds, 0.1, 1.0 and 10.0mm/min. Visual assessment of the specimens showed crazing in the region directly opposite the initial crack formation point i.e. the last part of the specimen to fail. Further microscopic evaluation showed small amounts of crazing in the immediate vicinity of the whole fracture line. The location of the crazing agrees with the literature, in that it is not visible in the pure tensile region of the loading, however, crazing is visible in regions of failure, where the stress field is not uniaxial. Due to the relatively low amounts of visible crazing and the small area over which crazing was evident this method was not further developed.

The second type of simulation was a simple three-point bend test, which is commonly used to assess environmental stress cracking of polymers.[55, 73, 91] Again, the three sample groups were used; control, DOS oil and wax. Samples of approximately 40 x 10mm were loaded into the apparatus, as shown in Figure 4.12. A known displacement was then applied to the central beam, causing curvature in the specimen. Samples were then unloaded and examined. Visible crazing was evident on the points of tightest curvature, and thus regions of maximum stress. Microscopic examination found that all crazes ran at 90° to the applied stress direction. However, the test did not provide a very good simulation of the dome-crazing phenomenon.

Both of the test methods investigated above apply simple uniaxial stress fields to the sample. Because the dome of a beverage can involves some form of biaxial stress, it was decided to investigate this process. In the third type of test a set of dome tooling was fitted to the Roell & Korthaus cupping machine and used to produce a simple dome in a flat disc. A blank holder force that allowed some material flow was chosen (9.8kN). It was also decided that no lubricant should be applied, thus avoiding any interaction with the polymer. Similarly, all identifying marks were located on the periphery of the sample, remote from the predicted crazing region, negating any effects of solvent based ink upon crazing performance. Initial dome simulations were formed to a depth just before complete failure. This method of biaxially stressing the material gave a crazing ring analogous to that seen previously on the problem cans. As a result this third method of crazing simulation was used as the basis of the following work. Further refinement of this deformation process resulted in the procedure highlighted subsequently in section **4.2.4**.

Measurement of stress whitening

Initial visual assessment of the crazing showed that the phenomenon was only produced at specific regions of the dome. In the majority of cases this was seen as a ring of crazes. Surface microscopy of the crazes showed them to have a grain-like structure of varying size and density depending on exactly where within the crazed region was analysed. Optical microscopy of these regions proved difficult due to the curvature of the sample and the resultant restrictions in depth of field. In an attempt to combat these restrictions the use of SEM analysis was considered. The low pressure

SEM at UWS was used to further investigate the crazed areas. Two images were taken from sites approximately 2mm apart across the crazed region (centre to edge), see Figures 4.14 and 4.15. It is clearly shown that in these two close regions, the size and extent of crazing is different. These differences are attributed to the rapid changes of major and minor strain over a small area within a biaxially stressed component.[92] This vast variation in craze dimension over a small area makes accurate measurement of crazing very difficult. Using a craze intensity or individual size measurement to quantify crazing in this case would produce large experimental error and produce problems when comparing results between different samples.

Photographic representation of crazing also proved troublesome. The light required to illuminate the surrounding areas disguised the capture of the crazed region. Ultraviolet inks provided a solution. The application of ultraviolet ink to the crazed region for a few seconds provided an ideal way of highlighting the crazed area. The ink was drawn into the crazes by a form of capillary action, but not into the non-crazed regions. Analysis of the samples under ultraviolet light clearly highlighted any crazed regions in the specimens. This allowed visual representation of the crazing, a selection of specimens is shown in Figure 4.16. Clockwise from the bottom right are the respective levels of crazing, from none to the most severe example.

Quantification of the crazing has proved difficult. Initially, it was decided to try to measure polymer breakdown as a function of porosity. Defects in can making can be detected as current flow through as electrolytic solution placed inside the container. Accurate readings can only be taken when metal exposure, through the polymer, results in high current flow. However, crazes by definition do not always penetrate through the thickness of the polymer, and hence do not expose the metal substrate. In fact, only the most severely crazed samples had adequate metal exposure to generate meaningful results. Therefore, this method could not detect partial crazing that does not extent across the coating thickness and is of little use for craze quantification measurements.

Because crazing density changes so rapidly within a small area, and measurement of metal exposure is inconsistent, another method of quantification was required. The simplest approach, with best results, involves measurement of the crazed area. This

was achieved by measuring the extent of the crazed matter both along and across the rolling direction and calculating the affected area. A schematic diagram of this approach is shown in Figure 4.17. Due to the elliptical nature of the crazed region, the following standard equation was used to calculate to whitened area ($Area_e$):

$$Area_e = ab\pi \qquad \textbf{Equation 16}$$

where 2a and 2b are the lengths of the major axis of the ellipse.

When only the periphery of the ellipse exhibited crazing then the difference between the crazed and unaffected areas was taken. In this way for any given sample measurement of the whitened region along both major axis (perpendicular and parallel to the rolling direction) results in generation of a quantifiable crazed area.

In biaxially stressed panels, deformation research regularly uses an etched grid marking system to measure component strain.[93, 94] This approach was investigated for the crazed dome simulations. However, a number of problems were encountered in analysing polymer coated steel. Firstly, the grid marking used in automotive research uses an electrolytic etching technique.[95] This method cannot be used for laminate materials in their coated form, due to the non-conductive properties of the polymer layer. Two other methods were investigated; the first grid marked the substrate prior to laboratory application of the polymer film. The second involved printing a grid onto the film prior to laminating. Both these methods suffered from changes in forming properties due to the ridged profile of the grid pattern. Additionally, the amount of deformation applied to the discs when simulating dome production was not accurately measurable by the facilities available. Further deformation of the samples to a point were the change in grid pattern was detectable could not be achieved, due to the low percentage elongation to failure of DR substrates used in steel polymer laminate applications. Therefore, this avenue was not explored further within this research programme.

4.2.4 Forming procedure

Dome simulation

The chosen 'dome simulation' method produced a small dome in a flat 125mm disc on the R&K cupping press using a set of dome tooling. The profile of the resultant dome simulation was controlled by manually adjusting the punch height against an electronic readout to an arbitrary value. Dome simulations were formed with depths ranging between 0.5 and 10mm. The total crazed area as a function of recorded dome depth is shown in Table 4.7 and Figure 4.18. A value of 6mm was chosen as the standard height for these experiments, because it gave sufficient amounts of crazed matter to be measurable, but did not fracture the material. For the materials investigated the sample failed by cracking along the region of maximum stress at dome depths greater than 9.0mm. This agrees with tensile data for this material, shown in Table 4.4, which shows elongation to failure of less than 2.0%. This dome was then compared against a standard specimen to ensure good reproducibility of results. The following is the standard operating procedure developed for the dome simulation experiments.

Machinery settings:

- ◊ 0.22 steel die
- ◊ Dome punch tooling
- ◊ High speed off
- ◊ Nitrogen bag accelerator lever closed
- ◊ Manual speed setting at zero

Experimental procedure:

1. Place sample in locator ring, rolling direction away from the operator.
2. Lower die and it's housing, locking handle into place by pushing away from operator. N.B. Machinery will not operate if micro-switch is not contacted.
3. Press both start buttons.
4. Set blankholder pressure at 1.5 tonnes.
5. Watch ram hieght display on the data box. Adjust speed dial until height of +6.00mm is achieved (± 0.05mm).

6. Press stop button.
7. Unlock the locating lever by pulling towards operator, lift the housing up and secure with locking clamp.
8. Remove sample from the machine. Mark sample with time and date of experiment, noting maximum ram height (if different from standard).
9. Compare sample with control, to ensure that all readings taken were accurate and that all machinery zero readings are correct and have not been altered by a previous operator.

Repeat from 1. For each separate dome simulation

Crazing assessment

As shown in section **4.2.4**, the only reliable method of quantifying the crazing was by measurement of the affected area, usually elliptical in nature. Calculation of the area was taken from measurement of the major axes, then using Equation 16, the extent of the crazing can be generated. In the case of incomplete crazed dome, the difference between the smaller and larger ellipses was used instead. This value then allows comparison of one disc with another.

4.2.5 Experimental investigation

The experimental work conducted to understand the crazing phenomenon was split into two areas; ageing and prestoving of film laminates prior to deformation.

Ageing effect of fresh plate

To more fully understand how the crazing phenomenon changes over time, it was decided to analyse the formation of crazes on a selected material at various time periods after lamination. The material chosen for this analysis was no. 2 in Table 4.6. Due to evidence that crazing magnitude is affected by preheat temperature of the Ferrolite line, three preheat variables were used: 165°C (typical of a PP laminating route), 215°C (a standard preheat PET laminating temperature) and 230°C (analogous to certain critical production routes). All other line variables were set as for the standard PET laminating cycle.

Material was tested by deformation under a biaxial stress field at various time periods after lamination and assessed for crazing occurrence and magnitude. Material no. 2 in Table 4.6 was also analysed by differential scanning calorimetry at time periods of 2 and 100 days after production. A specimen from the latter sample group was also heated to 95°C and rapidly cooled to room temperature. This sample was also analysed on the DSC apparatus. For all experiments, particular attention was given to the region around the glass transition temperature (T_g) of the laminate coating, approximately 75°C in PET films.

Prestoving experiments

Prior to these experiments, it was suspected that preheating the cup to approximately 80-90°C prior to dome formation (stoving) could remove the appearance of crazing. However, it was also known that if left for a period of time, crazing would subsequently reoccur. In an attempt to quantify this phenomenon, a series of experiments were carried out. These involved heating a flat disc to a set temperature for one minute, then forming the dome after various time intervals to see if or when crazing occurred. The oven temperatures chosen for this experiment were 60°, 70°, 75°, 80°, 85°, 90° and 100°C. At each temperature level, nine discs were tested. The chosen time intervals were 30 seconds, 2 and 5 minutes, 1 and 6 hours, 1 and 3 days and 1 and 4 weeks after stoving. At the shortest time intervals of testing the samples still retained some residual heat from the stoving experiments. However, when the stress was applied the temperature of the disc was lower than 75°C, the critical glass transition temperature for PET.

Rolling lubricants

It will be shown that the presence of certain rolling oils on the surface of the substrate prior to lamination affects the formation of crazes. Therefore, a method of producing a film laminated sample without any rolling oils was necessary to establish the full effect of any interaction between these chemicals and the polymer film.

Two methods were highlighted. The first was a laboratory simulation of the film application process. The second involved testing of a new grade of film laminated

material, which bypasses the ECCS coating line. This avoids the rolling oil application stages that occur at the end of that unit. The former method proved inconclusive due to the difficulty in producing specimens that exactly duplicated the line laminating conditions. The latter method was a preferred option because the material was produced on a commercial scale, for an intended end use. The laminate specification used was a 0.17mm DR620 blackplate grade with PET 20μm clear applied to one side and PET 25μm white to the other (material 2 in Table 4.6). This specification differs marginally from the material used as a standard for all the previous experiments. However, it offers subjective comparison to establish whether the removal of rolling oils alters the craze formation in laminated material.

Lubrication applied prior to deformation

As stated earlier in section **2.4.3**, it has been noted that the addition of wax, applied previously to facilitate drawing of the part, may affect the deformation behaviour of film laminated materials under a biaxial stress field. Therefore, it was decided that various lubricants should be applied to the surface of a sample and left for assorted time periods to see what differences, if any, occurred. Petrolatum based wax was tested along with lubricants applied to the strip prior to lamination, namely Dioxylsebacate (DOS), Acetyltributylcitrate (ATBC) and Butylstearate (BSO). These lubricants were applied in a thin film to the samples, all excess was removed, and the samples were left at room temperature for 1, 10 and 28 days prior to formation. The fresh plate had lubricants and waxes applied to the surface within 24 hours of lamination. Therefore, one day must be added to the testing times to compare directly with the work on analysis of ageing of laminate material.

4.3 Ancillary testing methods

4.3.1 Optical microscopy

Standard optical microscopy of film laminated materials is slightly different to that conducted for packaging grade steel analysis. The sample must be cold mounted because the hot mounting procedure causes thermal damage to the polymeric film. For cold mounting a ratio of 15:2 for resin and hardener is sufficient for most requirements. If it is necessary to examine the clear laminated coating a few drops of a dark dye must be added to the mounting solution. This ensures sufficient contrast between polymer and the surrounding mount under reflected light conditions. Grinding and polishing procedures are very similar to those employed for hot mounted steel samples. However, more care must be taken because the resin is softer and requires less time under pressure to acquire the necessary surface preparation.

4.3.2 Scanning electron microscopy

SEM analysis of crazed material was carried out on the low pressure apparatus at the University of Wales Swansea. Circular samples of approximately 25mm in diameter were coated in a thin layer of gold by vapour deposition prior to analysis. Examination of the crazed area was then undertaken at a magnification of x350, with an accelerating voltage of 10kV. Suitable pictures were then digitally captured for further analysis.

4.3.3 X-ray photoelectron spectroscopy

XPS is a technique that allows qualitative and quantitative assessment of a sample surface. X-rays of a known energy are fired onto a sample and the resultant photoelectrons, which are emitted, relate to specific binding energies of particular atoms, chemicals or bond types. Knowledge of the position and magnitude of the binding energy peaks allows chemical analysis of the first few atomic layers of a sample. All data reported in this document using XPS analysis was conducted by trained personnel at Corus' Swinden Technology Centre.

4.3.4 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is a technique used to determine enthalpy changes within examined materials. Typically two samples are heated concurrently; the first is a reference. The second is the investigated. The samples are heated at a constant rate until a transformation point is reached. At this point the sample heating rate is increased or decreased to compensate. The results are then plotted as heating rate against temperature. Any changes from the underlying trend represent a transition point of the investigated material. The DSC apparatus can be used under heating and cooling cycles, but the latter is less accurate.

4.3.5 Cup wall profiling

On occasions it was important to understand how the wall profile of a cup varied up its height. This was achieved by removing a portion of the cup and measuring the gauge thicknesses of the sample at 5mm intervals. For each quoted cup wall profile, two samples were measured three times at every 5mm interval from the base of the cup. Note was also taken of the starting gauge in the base of the cup because cup wall comparisons are clearer if quoted as a fraction of the original gauge.

4.3.6 Friction measurement

Certain materials investigated within this document were the subjects of frictional coefficient assessment. This was accomplished on an Altek machine. A 2kg weight supported on three ball bearings was dragged at a constant force across the surface of the specimen. A strain gauge measures the variation in force with time, which can be related to the frictional co-efficient of that material condition. The resultant measurements could then compared with other materials assessed in the same manner.

5 Drawing of Film Laminated Materials

5.1 Results

Unless otherwise specified all cupping data is shown as average minimum height, clear coating internally, formed using tooling set B in Table 4.2. The clear coating inside represents the standard industrial process route. The 2.0mm die radius represents a die radius to material thickness ratio of 7:1 – 10:1 for most materials investigated within this programme, an optimum value suggested in literature, which allows production of consistent repeatable results.

5.1.1 Effect of substrate properties

Substrate passivation

Substrate mechanical properties for 0.195mm and 0.26mm thickness materials are shown in Tables 5.1 & 5.2 respectively. The average proof stress for the 0.195mm substrate was 585.4MPa and showed 10MPa variatons across the width of the strip (edge to centre) and 5MPa along the strip. The average ultimate tensile stress (UTS) was 588.3MPa with variations of 6MPa noted in both directions. The observed variability equates to less than 2% difference within both ultimate tensile stress and proof stress data values.

The 0.26mm material shows an average proof stress of 589.0MPa, with variation of less than 5MPa both across and along the sheet samples. The average UTS was 592.6MPa, with 5MPa variation in both directions. This observed variability represented less than 1% difference in UTS and proof stress properties. For both materials the elongation in all cases was less than 2%. The above proof stress, UTS and elongation values are all well within the acceptable range of mechanical properties indicated in Table 5.3, thus confirming that the substrate materials were typical of a steel grade for deep drawing applications.

Tables 5.4 and 5.5 show the failure rates for each of the three coating types, for 0.195mm and 0.26mm substrate respectively, in the variable blank holding force experiments. Failure rate for blackplate substrate was 52% (0.195mm) and 60%

(0.26mm). This compares to a rate of less than 5% for both remaining coatings at 0.195mm and rates of 20 % and 3% respectively for 0.26mm lacquer and 0.26mm film laminated material.

Figure 5.1 shows a comparison of minimum cup height values for each of the three coating variables on the 0.195mm substrate at three blank holder forces. It is clearly seen that the blackplate samples are higher than the two other variables. At 14.7 kN blank holder force the blackplate material showed an average height of 42.54mm, compared to 42.19 and 41.96mm for the lacquered ECCS and film laminated conditions. For each condition cup height increased proportionally with blank holder force.

Figure 5.2 also shows a comparison of minimum cup heights for each of the three coatings, but this time for the 0.26mm substrate. The results are very similar to the previous graph, with blackplate cups being taller than both lacquered ECCS and film laminated samples, with little difference between the latter two variables at 14.7kN blank holder force. The blackplate condition showed a cup height of 43.56mm, whilst the lacquered ECCS and film laminated variables showed heights of 43.01 and 43.12mm respectively.

For each surface condition the co-efficient of friction was measured on an Altek machine, using a 2kg weight. The results are indicated in Table 5.6. The 0.195mm substrate gave average values of 0.362, 0.117 and 0.103 for the blackplate, lacquered ECCS and film laminated samples respectively. This shows a decrease in co-efficient of friction when either coating is applied relative to the uncoated blackplate stage. The 0.26mm substrate material shows a similar magnitude of friction for each coating type, namely 0.291 (blackplate), 0.164 (lacquered ECCS) and 0.121 (film laminate). In both cases the range between maximum and minimum values was greatest for the blackplate material.

Substrate thickness

Comparisons of the minimum cup height of four film laminated materials of various gauge with nominal tensile strength of 620MPa are shown in Figure 5.3. Linear

increases of height with increasing gauge are noted when forming over both 1.0 and 2.0mm die radii.

Tensile data for all four substrates is shown in Tables 5.1, 5.2, 5.7 and 5.8. They represent the specimens taken from centre and edge positions within the tested region. Tables 5.1 and 5.2 have been commented on in the previous subsection. Tables 5.7 and 5.8 show an average ultimate tensile strength of 589.1MPa and 592.1MPa for the 0.15 and 0.24mm gauge substrate. This increase in strength with thickness is not significant in relation to scatter of the data. In both cases the variation in tensile results across and along the tested region was <20MPa.

Substrate strength

Tables 5.1, 5.9 & 5.10 show mechanical properties for three different substrates of 0.19mm nominal thickness. Table 5.9 shows an average proof stress of 501MPa for the DR520 material. The samples exhibit a variation of 5MPa along the strip and 10MPa across the width. The average ultimate tensile strength (UTS) is 505MPa, with 10MPa variation in both directions. The observed variability in both UTS and proof strengths is less than 2%. Table 5.10 (DR580) shows the same format of mechanical properties as Table 5.9. Proof and UTS measurements average at 559 and 562MPa, with variations of 10MPa across the width and down the length of the investigated area. This equates to a 1.8% difference in strength properties. Table 5.1 similarly shows UTS and proof strength readings for the last material investigated in this analysis (DR620). Average values are 585 and 588MPa respectively and exhibit variation of 15MPa in both directions (2.6%.). All three variables considered above have proof stress, UTS and elongation values well within the acceptable limits for DR520, DR580 and DR620 materials as shown in Table 5.3.

Figure 5.4 plots the minimum cup height of a range of PET film laminated materials of 0.19mm nominal thickness against each material's measured average UTS. It is clearly shown that as substrate strength increases, cup height increases in a non-linear fashion within the investigated range.

5.1.2 Effect of polymer coating

Polymer type

Comparison of two film laminate materials with identical substrate specifications, but differing polymeric coating type is shown in Figure 5.5. Materials no. 12 and 14 from Table 4.1 were formed over a 1.0mm die radius at a range of blank holder forces. At the lowest blank holder force considered (14.7kN) the respective cup heights for the polyethylene terephthalate and polypropylene film laminated materials were 39.24 and 39.22mm, a difference of 0.06%. At the other extreme of the operating window (44.1kN) the PET and PP cup heights were measured at 40.30 and 39.89mm respectively, a difference of 1.03%. For both coating types the increase in cup height with blank holder force was approximately linear.

Polymer thickness

The influence of polymer thickness on the drawn cup height of a disc was considered for both polypropylene and polyethylene terephthalate. Minimum cup height over a 1.0mm die radius for a range of blank holder forces for each polymer type is shown in Figures 5.6 and 5.7 respectively. The PP coated laminate shows greatest difference between the 20 and 40μm materials at a blank holder force of 24.5kN. Cup height exhibits average minimum values of 38.58 and 38.53mm, respectively, a difference of 0.13%, which is within the spread of the data. Figure 5.7 considers the PET laminated material. This shows a maximum difference between the 15 and 30μm coatings, at a blank holder force of 44.1kN, of 0.09mm or 0.23%. Again, this difference lies within the measured data scatter. In both cases cup height increases proportionally with increasing blank holder force.

Polymer pigmentation

The effect of coating pigmentation on drawn cup height is shown in Figure 5.8. A material, with white and clear PET films on opposing sides of the laminate was formed under conditions where half of the sample set was produced with the clear coating on the inside of the cup of the laminate in contact with the die during forming. Conversely, the other half was produced with the white coating on the inside of the cup. The greatest difference noted in measured cup height was at 34.3kN blank holder force. The white pigmented coating shows an average minimum cup height of

38.69mm, compared to 38.54mm for the unpigmented (clear) coating in contact with the die during forming. This represents a difference of approximately 0.39%.

X-ray photoelectron spectroscopy (XPS) was used to determine the chemical composition of the first few atomic layers of the white pigmented and clear laminated material. This is shown in Table 5.11. In both cases the Carbon and Oxygen combined exceed 99.7% of the total. Titanium, used in the pigmentation process, was below the detection level of the machine (0.1%) for both polymer colours considered in this analysis.

Atomic force microscope (AFM) imaging of a representative $5\mu m^2$ sample of each polymer colour is shown for white pigmented and clear material in Figures 5.9 and 5.10. A three-dimensional image of coating topography is visible. The white surface shows a number of ridges within the examined area. Conversely, the clear coating exhibits far less undulations in same the magnitude of investigated region.

Laminating conditions

Materials 14 and 16 in Table 4.1 have the same polypropylene film laminate specification, but were laminated under different temperature regimes. These represent the standard PP (low) and PET (high) laminating conditions. This has been introduced in more detail in section **4.1.5**. The average minimum height of the drawn cups is shown over a range of blank holder forces in Figure 5.11. Maximum difference between the laminating conditions data series is at a blank holder force of 34.3kN. The high and low data series show cup heights of 39.99 and 39.67mm, a difference of 0.81%. It was also notable from the experiments that cups produced at the higher blank holder forces were more likely to exhibit top-wall wrinkling. This was true for both materials investigated.

5.1.3 Effect of tooling

Blank holder force

Figure 5.12 shows how minimum and maximum cup heights for material no. 12 in Table 4.1 vary with blank holder force. It is clear that both measured values of cup

height increase linearly with increasing blank holder force. This agrees with data shown in previous figures. However, the minimum and maximum cup data series are not parallel. Therefore the difference between the two readings (earing or Δh) decreases. At a 14.7kN blank holder force the earing value is 3.77mm. At 539.kN blank holder force this earing value has decreased to 3.29mm. Percentage earing values are 9.94 and 8.49% respectively.

A comparison of cup wall profiles from the extremes of the operating window examined in Figure 5.12 are shown in Figure 5.13. The cup produced at the largest blank holder force shows more wall thinning than the cup produced at the lower blank holder value. Respective wall thickness at 5mm along the cup wall is 0.205 and 0.198mm. This compares to an original gauge of 0.225mm. As a fraction of original gauge, wall thickness varies non-uniformly from around 0.9 to over 1.3; a 30% increase in cup wall profile.

The operating window for all laminates produced from the 0.18mm DR580 substrate is shown in Figures 5.14 and 5.15. These represent the operating windows for 1.0 and 2.0mm die radius respectively. In both cases the PET coatings have the largest operating window (40kN). The PP (clear) coating has the smallest operating window, 25kN at 1.0mm die radius and 30kN at 2.0mm die radius. Increasing die radius from 1.0 to 2.0mm results in a vertical shift for the minimum blank holder force of 10kN for materials widely used in commercial applications.

Die radii

A set of experiments was conducted to assess the influence of die radius on the cup height of film laminated materials. Figure 5.16 shows how laminate number 12 in Table 4.1 reacts to changes in die radius. Cup height decreases with increasing die radii. At 0.75, 1.0, 1.36 and 2.0mm die radius, cup height averages are 40.51, 39.66, 39.03 and 38.17mm respectively. Variability in these data sets is approximately 0.5mm. Figure 5.17 shows how cup height changes with die radius for three cut edge dimensions; 125, 120 and 100mm diameter. Again, each data set shows the same shape curve regardless of cut edge dimensions. Subsequent analysis of the cup wall profiles for the 125mm discs is shown in Figure 5.18. A representative cup from each

data set is shown as a percentage of its original thickness (0.225mm) in relation to height along the cup wall. All three cups show a degree of wall thinning (fraction of original thickness <1). Additionally, all samples also exhibit wall thickening towards the top of the cup. This approaches 30% for the 2.0mm die radius cup. The amount of lower wall thinning is slightly greater at the tighter die radii. The tightest die radius considered 1.0mm has a wall profile that remains less than its original gauge until 25mm up the cup. Increasing die radius to 2.0mmm results in a wall profile that is negative for only the first 15mm of the cup height.

Figure 5.19 shows how minimum cup height varies with blank holder force over two die radii; 1.0 and 2.0mm, using material no. 12 in Table 4.1. An approximate linear increase is shown in both data groups. At all blank holder forces considered the 1.0mm die radius data draws a taller cup than the equivalent values from the 2.0mm die radius data range.

Cut edge

A comparison of cup height over the range of cut edge dimensions is shown in Figure 5.20. A linear relationship can clearly be seen with larger cut edge dimensions increasing the cup height. Three die radii are also considered and with all data series, the tighter die radius draws a taller cup. Extrapolation of any of the three series back to zero cup height (x-intercept) indicates a value of approximately 72mm. This compares to a punch diameter of 67mm for these experiments, a difference of 7.5%.

Measurements of cup wall profiles for the 1.36mm die radius are shown in Figure 5.21. In all cases, wall thickness increases up the cup. For the 100mm cut edge disc, the fraction of the original thickness is always >1. The remaining two cut edge dimensions have a lower wall thickness, less than the original gauge. At some point, midway up the cup wall, the fraction of original thickness becomes >1 and continues to increase until the perimeter is reached.

To investigate the influence of cut edge diameter on the drawing characteristics of film laminated materials, four disc sizes were considered, 100mm, 120mm, 125mm and 135mm. However, it was not possible to manufacture suitable cups at largest cut

edge diameter. Limited cups were produced, but top wall wrinkling was so severe that accurate height measurement was impossible and the likelihood of permanent tooling damage sufficient to discontinue the experiment.

Punch diameter

The effect of punch diameter on cup height for a given cut edge is shown in Figure 5.22. At a cut edge of 125mm the minimum cup height measured for punch diameters of 66.79, 88.92 and 90.40mm were 38.17, 21.15 and 20.55mm. These values for a 120mm cut edge were 34.57, 17.87 and 16.95mm respectively. The change in cup height with cut edge for each punch diameter is linear. However, not all the data series are parallel to one another.

5.1.4 Effect of lubrication

Lubrication weight

A number of identical discs of material 1 in Table 4.1 were manufactured under the standard operating procedure. Each disc had a different lubrication weight applied. This ranged from 0.002 to 0.05g per disc. The effect of lubrication weight on cup height is shown in Figure 5.23. The experiment shows that cup height decreases with increasing lubrication. Between the two extremes, cup height varies from 40.08 to 37.48mm, a difference of 7%. Change in cup height is more noticeable at very low lubrication weights. Conversely, at higher lubrication weights the difference is minimal.

Lubricant type

The second area of investigation analysing lubrication type considered four different commercial alternatives; petrolatum wax, Valspar, EZ11 and a control, the oil isopropanol alcohol used throughout this document. Fifteen discs of material 12 in Table 4.1 were used for each lubrication type. Approximately 0.75g of each lubricant was applied to each disc set. Samples were then cupped using the standard operating procedure.

Figure 5.24 shows the average minimum cup height of fifteen discs produced different lubricant types. For the four types of investigated lubricant, petrolatum wax, Valspar, EZ11 and the control average minimum drawn cup heights were measured at 39.12, 38.90, 38.89 and 40.00mm respectively. This represents a total difference in cup height between the shortest (EZ11) and tallest (control) average values of 2.85%.

5.2 Discussion

This section of the experimentation investigates the forming characteristics of film laminated materials when drawn into a cup from a flat disc. Section **2.4** of the literature survey highlights the critical factors that govern formation of this first stage draw. Of these areas the following experiments were conducted to investigate how substrate, coating and tooling issues alter forming characteristics. Additionally, the influence of lubrication type and weight was also examined.

5.2.1 Effect of substrate properties

Film laminate materials are produced on a range of substrates. Their thickness can vary from 0.1 to 0.4mm and have a nominal ultimate tensile strength of between 480 and 620MPa. As discussed in section **2.2.3 – 2.2.6**, the substrate can be batch or continuously annealed and have single or double reductions. Prior to application of the polymer film at the laminating line the substrate can be treated/passivated in two ways; electrolytic tinning or electro-chromium coated. Alternatively, the substrate can be left uncoated in a 'blackplate' form. However, in the last case, some passivation of the oxide layer is required for successful lamination, this is usually achieved by addition of a silane/zirconium coating.

This section of work investigated how different specification substrates alter the forming characteristics of film laminated products. The three most significant highlighted from the literature or of most commercial importance are coating/passivation, thickness and strength.

Substrate passivation/coating

To investigate how different substrate passivation influence forming characteristics a number of variables were considered:

- Uncoated blackplate material
- Lacquered electro-chromium coated steel
- Typical film laminate specification, 25μm white and 20 μm clear PET.

Each passivation treatment was applied to two gauges; 0.195 and 0.26mm, representing a wide range used for commercial can making. They had a DR620CA specification, from a 3165 low carbon aluminium-killed steel grade. Comparisons of cup height and frictional properties were then undertaken between each variable.

Mechanical properties for packaging steels such as proof stress, UTS and elongation are controlled to required limits. Typical values for a DR620CA material are shown in Table 5.3. Clearly, high quality sheet material is required to ensure the required dimensioned can is produced by the subsequent forming operations and that the performance of the finished product is acceptable. The results for the present tests shown in Tables 5.1 & 5.2 confirm that the substrates investigated in this document are representative of this specification material.

It is a recognised phenomenon that most steel produced in strip form has a variation in mechanical properties from centre to edge and across the width of a coil. These differences are mostly due to the variation in cooling behaviour of the centre and periphery of steel slab during hot rolling, resulting in edge samples being stronger than centre samples. This has been demonstrated by Jenks,[90] who showed an edge to centre variation of 25MPa for both proof stress and UTS in a typical double reduced steel plate used for DRD applications. This reasoning explains the variation in mechanical properties identified in Tables 5.1 and 5.2. The small variation in substrate mechanical properties shown in these tables indicate that material variation for both thicknesses is lower than usual. However, this is to be anticipated because the Ferrolite production route trims a small amount off the edge of each coil after lamination, to ensure that the polymer coating extends to the periphery of the coil. This reduces edge effects in the substrate coil and explains why the variability in tensile properties is less than in other typical examples.

The majority of the analysis was conducted at three distinct blank holder pressures within the blank holder force operating window of the materials investigated. The three values were determined by investigating the minimum and maximum values of clamping force achievable for the film laminated material between excessive wrinkling and tear-off. All three coating states analysed on both substrate variables showed that increasing blank holder force increased cup height.

Interestingly, failure rates within the three coating states varied greatly, shown in Tables 5.4 and 5.5. The uncoated blackplate samples on both substrate thicknesses showed over a 50% failure rate, with failures occurring at both high and low blank holder forces. For both substrate thicknesses the lacquered surfaces exhibited significantly less failures than the blackplate, but more than the film laminated samples. However, in both the lacquered ECCS and film laminated conditions, failure was limited only to the higher blank holder forces. This phenomenon was also reported by Shang *et al*,[96] who attributed the higher incidence of failures to over-stretching of the blank during forming. The increasing likelihood of failures at higher blank holder loads indicates that the material is approaching its forming limits at this particular draw ratio. This is to be expected because the upper blank holder force was specifically chosen to be near the extremities of these forming limits to ensure the widest range of blank holder pressures could be investigated. It does therefore show that the forming limit of a sample in deep drawn applications is not at a definite point, but occurs over a range.

Figures 5.1 and 5.2 indicate that the blackplate material, when successfully cupped, achieved a greater cup height than the two coated alternatives, by a value of approximately 0.4mm. This value is well above the standard deviation of the results. This data agrees with the information shown in Table 5.6. This shows that the uncoated blackplate material has a higher coefficient of friction than either of the two coated states. It is reasonable to expect that a material with a high coefficient of friction has a larger amount of retardation over the die radius; thus producing a taller cup. This relationship agrees with a study, which examines the relationship of frictional coefficient between blank and tooling and resultant cup height.[97] Investigation of the lacquered ECCS and film laminated coatings through the range of blank holder forces shows different results for the 0.195mm and 0.26mm substrates. In the former the lacquered material forms a cup that is 0.15mm taller. In the latter case the opposite was true, the lacquered cups were 0.07mm shorter than the film laminated alternatives. In both cases the spread of data is larger than the difference and it is difficult to conclude whether the different coatings affect the forming characteristics of the material between the two investigated states. The main reason for the differences between the forming of the coated and uncoated sheet is due to

frictional effects; this is shown in Table 5.6. The uncoated material had a significantly higher co-efficient of friction than the lacquered ECCS and film laminated variables. This explains why fewer failures were exhibited on the coated materials. The film laminate material justifies its existence on failure rates alone. In all cases it had the lowest failure rate. This would suggest a reduction in machinery downtime on a commercial application. However, literature states that failure rates decrease with increasing punch speed,[96] explaining why these problems are not as commonly encountered at commercial can making speeds, which are many times greater than those produced on laboratory equipment. Nevertheless, tool life would increase because every failure marginally damages the tooling and any stoppage is undesirable. Therefore any increase in productivity would be welcomed. Further work on the effect of forming speeds would be required to assess whether these effects are valid.

Substrate thickness

Four film laminated materials of 620MPa nominal strength were formed over two different die radii; 1.0 and 2.0mm. Minimum cup heights for each experiment are shown in Figure 5.3. Clearly both sets of die radius data cause a linear increase in cup height with substrate gauge within the range investigated. This relationship is attributed to a proportional increase in substrate gauge, which enables more material volume to be drawn into a cup. Cup height (in mm) can be expressed as a function of substrate original thickness (T_i) in Equations 17 and 18.

1.0mm: $h = 16.5Ti + 36.85$ **Equation 17**

2.0mm: $h = 16.5Ti + 36.9$ **Equation 18**

There is a 1.75mm decrease in cup height when increasing die radius from 1.0 to 2.0mmm. Both the above equations are only valid for substrates of 620MPa nominal strength and laminated with 20μm clear and 25μm white PET coatings.

It is expected that the change in cup height would remain linear for all substrate strength values. However, there is no guarantee that the gradient of the best-fit line would remain unaffected by changes in substrate strength. Further work on a wider range of laminates would be necessary to validate this assumption.

Substrate strength

Three substrates of nominal thickness of 0.19mm were laminated with the same specification polymer coating (20μm clear and 25μm white PET) and cupped using the standard experiments adopted previously. The variation in cup height was then noted and analysed with respect to the substrates ultimate tensile strength. The substrates in question show good consistency in both mechanical properties and grain size measurements. Although the actual average UTS and proof strength were lower than their nominal values they were well within the acceptable limits set in the quality documents, summarised in Table 5.3.[98]

Figure 5.4 shows how the forming characteristics of each of these film laminates changes with respect to substrate average ultimate tensile strength values. It is reasonable to expect cup height to increase with increasing substrate strength because a stronger material provides more resistance to bending across the die radius. The relationship between stronger material and taller cups was also shown in the variability in material properties section, used in development of a standard testing procedure (section **4.1.4**). However, the relationship would not be expected to be linear, otherwise strength could be increased continually to achieve taller cups. The cup production seems to be approaching a limiting value at the upper end of the investigated substrate strength range. This assumption is backed up with consideration of the data presented in Figure 5.2 and Table 5.5. Cups produced at 0.26mm gauge and with a nominal strength of 620MPa were more likely to fail during drawing at higher blank holder forces than at lower blank holder force or thinner gauge. This was attributed to onset of an upper forming limit range. Therefore, substrates of nominal strength in excess of 620MPa for a gauge of 0.19mm must also be approaching an upper forming limit. This helps explain why the best fit curve of the data in Figure 5.4 is increasingly steep at higher substrate strengths.

5.2.2 Effect of polymer coating

Film laminate materials are produced with either polypropylene or polyethylene terephthalate coatings, with a typical thickness range of 15 to 40μm (although films of up to 200μm have been manufactured) and can be laminated under a range of

temperatures. This section of work investigated how these coating specifications; polymer type, thickness, pigmentation and production temperatures alter the forming characteristics of film laminated materials.

Polymer type

The influence of polymer type on the drawing characteristics of film laminated materials has been assessed by comparing polyethylene terephthalate (PET) and polypropylene (PP) cupping behaviour. This was achieved by cupping both materials at a range of blank holder forces over a 1.0mm die radius. The results of this experiment are shown in Figure 5.5. Both the PET and PP film laminated materials exhibit a proportional increase in cup height with increasing blank holder force. This agrees with data from previous experiments. However, the polypropylene film laminated material does not draw a taller cup than the PET coated material per unit increase in blank holder force. The difference in minimum cup height at the extremes of the operating window between the coating types is in the region of 1%.

The small difference in forming characteristics related to changes in film coating type could be due to many factors. The most obvious is experimental error. However, this is unlikely. Firstly, an average of 15 cups was taken for each data point, minimising the spread in results. Secondly, the PP coating draws a shorter cup by an increasing magnitude with each investigated increase in blank holder force. If this trend is film coating type related, what are the possible reasons? Subjective evidence of formed cups shows that the PP coating is more likely to exhibit wrinkling upon drawing, especially at higher blank holder loads. This suggests that the PP material causes problems in applying an effective blank holder load (since insufficient blank holder loads cause excessive wrinkling). This holds true when we consider film to film friction coefficients of both polymer types. PET has a higher value than PP.[99] Additionally, PP has a lower yield strength, typical in the region of 29-38MPa.[99] Therefore, PP will provide less resistance to forming and may exhibit local yielding at higher blank holder forces, assisting top wall wrinkling of the cups. These factors help show that PP coated materials are more difficult to draw than PET coated alternatives.

Polymer thickness

Both the polymer types considered throughout this document have been examined at two thicknesses. The four materials used for this analysis are numbers 1, 13, 14 and 15 in Table 4.1. The polypropylene variables (13 and 14) were laminated on the industrial line and compare 20 and 40μm clear coatings. The polyethylene terephthalate variables both originate from material no. 1. However, the thicker coated variable (15) had a double coating, laboratory laminated to each side. This also allows comparison of 15 and 30μm clear coatings. In addition to the standard cupping experiments, the range of blank holder force (operating window) through which a cup can be successfully produced was also noted.

For all blank holder forces the measured minimum cup height varied by a very small amount, <2.5%. This is a little surprising, because thicker polymer coatings were expected to alter the forming characteristics of the film laminated materials due to the change in the ratio of coating to substrate. Increasing the PP polymer thickness from 20 to 40μm on either side increases the coating percentage of the total laminate thickness from 18.2 to 30.8%. However, on the basis of the experimental results this change in coating percentage appears not to alter the drawn cup height. Secondly, there were concerns that a change in the die gap (D_{gap}), might affect the forming results. All materials were drawn through the same internal diameter die (D_{die}); this effectively decreases the die gap (D_{gap}) between to two laminate materials from 0.13 to 0.09mm. The die gap is defined as the difference on each side of the punch between the die and the sample at its original thickness. This is shown more clearly in Equation 19.

$$D_{gap} = \frac{(Ti - D_{die})}{2} \qquad \textbf{Equation 19}$$

However, the results also suggest that the change in die gap does not influence the drawn cup height. It is of course possible that the above two theories cancel each other out, but this is unlikely. Further work would be required on a wider range of laminate coating thickness' to assess which theory is more likely. Alternatively, a

wider range of tooling could be examined with identical die radius, but with increasing die gap.

Polymer pigmentation

A number of different laminate specifications, which have white and clear coatings on opposite sides of the material, exhibit very similar cup height properties regardless of which coating colour is in contact with the tooling during forming, Figure 5.8. Initial consideration of this phenomenon may find these results a little surprising. The white polymer is manufactured by addition of hard titanium dioxide particles ($Ti0_2$), to pigment the coating to the required colour. A typical $Ti0_2$ addition would be in the region of 12.5%. It would therefore be reasonable to assume that an interaction of these particles and the tooling during cupping would affect the laminates forming characteristics.

Surface topography of laminated PET analysed using an Atomic force microscope (AFM) shows a marked difference between the white and clear coatings, Figures 5.9 and 5.10. The white surface has a significantly greater number of ridges than the clear alternative. This is attributed to the presence of the titanium dioxide particles. However, analysis of the coating surface chemistry by X-ray photoelectron spectroscopy (XPS) is shown in Table 5.11. This technique allows compositional analysis of the first few atomic layers of the polymer coating. Both colours exhibit a titanium content lower than the detection limit of the apparatus, 0.1%. Therefore, it can be stated that no $Ti0_2$ pigmentation particles protrude from the surface of the white laminate coating. The analysis shows that the $Ti0_2$ has either 'wetted out' from the surface of the coating or is covered by at least a thin film of bulk polymer.

It is the XPS data, which explains why the cupping results show no difference between the forming characteristics of clear and white pigmented laminate coatings because it shows the consistency of surface composition of the laminate material. It is also reasonable to assume that the differences in surface topography are minimised by a combination of lubricant and applied blank holder force during forming. Additionally, the above reasoning remains valid for consideration of the

polypropylene laminate coatings and can be used to explain the same trends shown with the polyethylene terephthalate material.

Laminating conditions

There is evidence to suggest that laminating conditions affect the formability of polymer coated materials.[100] Concerns have arisen that the large temperature differences in the processing of film laminated polypropylene materials may affect their forming characteristics. Therefore, the same material was produced at the extremes of the acceptable production window and cupping characteristics compared. It is these two temperature regimes, which are used for materials 14 and 16 in Table 4.1. There is a window of acceptable preheat, postheat and soak temperatures, which produce successfully film laminated materials. In the case of polypropylene the usual values for the three critical temperature regimes are 160°, 220° and 200°C respectively. However, in circumstances where PET and PP are laminated onto opposite sides of a substrate, higher temperatures are required to achieve successful PET adhesion, typically 165°, 260° and 230° are used for the preheat, postheat and soak zones respectively.

Figure 5.11 shows how cup height varies for the two PP temperature regimes over a range of blank holder forces when produced over a die radius of 1.0mm. The maximum difference between the two laminate materials is at the highest blank holder force. However, excessive wrinkling occurs on these cups and causes a larger variation in results. This is attributed to the thicker polymer coatings interfering with the effective blank holder values. The maximum variability in the remainder of the data points is 0.32mm at 34.3kN blank holder value; a difference of less than 1%. The difference in cupping characteristics between the two laminating temperature regimes is smaller than the spread in the data and is not discernible from the natural scatter in the data. Therefore, the current temperature ranges are not large enough to cause great differences in the forming characteristics of PP coated materials under the present forming tests.

5.2.3 Effect of tooling

Blank holder force

The blank holder force required to produce a successful cup must be sufficient to eliminate excessive wrinkling, but not so high that failure occurs through tear-offs. These three outcomes are clearly visible in Figure 2.13. The range of blank holder force through which a successful cup is produced is named the operating window. Most literature agrees that the blank holder force should be set just high enough to eliminate trace wrinkling, although other sources have analysed a range of values within the operating window.

In considering the standard PET coated material, clear coating internally over the 2.0mm die radius, it is clearly seen that increasing blank holder force increases cup height (Figure 5.12). The trend in behaviour noted is reasonable because increasing the blank holder force increases the retardation force over the die radius during forming, thus inducing a greater degree of stretch in the material at higher blank holder forces. This agrees with data reported by Shang *et al.*[96] The assumption is confirmed by considering wall profiles of cups taken from each blank holder force examined. Figure 5.13 clearly shows that cups produced at higher blank holder forces have more wall thinning in the lower regions of the cup than the lower blank holder equivalent. This relationship extends throughout the cup wall, with thinner material always present at the higher blank holder force. The other notable point raised from Figure 5.12 is that the maximum cup height, which increases with blank holder force, does not increase by the same amount as the minimum heights. This has two benefits. Firstly, by using a higher blank holder force, taller cups area produced for the same cut edge dimensions. Secondly, because the difference between minimum and maximum cup heights, ΔH, decreases with increasing blank holder force, the amount of material lost through trimming at a later stage is also decreased. The possible downside of increasing the blank holder force and producing taller cups is that in subsequent redraws, required in can making, there may be a change in the percentage of failures. This work has focused entirely on the first stage drawing of laminate materials and it is therefore not possible to comment definitively on further redraw conditions.

The operating window for a given laminate material depends on the coating applied and the die radius over which it is formed, shown clearly in Figures 5.14 & 5.15. Looking generally at the data, the lower end of the operating window increases with increasing die radius. Additionally, the magnitude of the window also increases with increasing die radius, by having a higher upper limit. These trends are acceptable because less energy is lost over slacker die radius. Therefore, less blank holder force is required to suppress wrinkling when forming a cup over a tighter die radius.

Die radii

Figure 5.16 shows how cup height changes for a 125mm cut edge of material 12 in Table 4.1 when formed over four different die radii; 0.75, 1.0, 1.36 and 2.0mm. It is shown that cup height is greatest at tightest die radius. As die radius increases cup height decreases in a logarithmic relation ship described by the following equation:

$$h = -2.34\ln(Drad) + 39.66 \qquad \textbf{Equation 20}$$

The basic shape of this relationship remains similar for a range of cut edge dimensions shown in Figure 5.17. In all cases cup height is tallest when cupped in conjunction with the tightest die radius and shortest with the largest die radius. These trends are acceptable, a tighter die radius requires more force to bend and unbend the sample over the die profile in comparison to a larger die radius. This results in a greater degree of lower wall stretching and generates a taller cup. This theory is supported by data presented in Figure 5.18. Wall profiles from the 1.0, 1.36 and 2.0mm die radius experiments are shown as a fraction of their original thickness. The tighter die radius experiments have the thinnest cup wall at 5mm and also have greater proportion of the entire wall length with a gauge less than the original thickness. The suggestion of greater energy losses over tighter die radii is also reported in the literature.[76] This source suggest that die radius should be as large as possible to ensure less energy is lost during formation of the cup.

Figure 5.19 shows how cup height over a range of blank holder forces changes with two die radii; 1.0 and 2.0mm. In both cases the best-fit lines are parallel and can be described by the equation type $y = mx + c$. The approximate evaluation of these two data sets are shown in Equations 21 & 22.

$$1.0\text{mm: } h = mx + 39.35 \qquad \textbf{Equation 21}$$

$$2.0\text{mm: } h = mx + 37.6 \qquad \textbf{Equation 22}$$

Therefore, changing die radius from 2.0 to 1.0mm results in an increase in cup height of 1.75mm. This agrees with the data reported in Figure 5.3, which also shows a 1.75mm change in cup height when changing from a 1.0 to 2.0mm die radius.

Cut edge

To analyse the effect of cut edge on cups drawn from film laminated materials, four different diameter discs were formed over three die radii, 1.0, 1.36 and 2.0mm. The standard PET film laminated material, no. 12 in Table 4.1 was used for all experiments. It has been shown previously (section **5.2.1**) that the material used in this investigation has consistent mechanical properties within an acceptable range for typical applications.

Figure 5.20 shows how increasing cup edge dimensions increase cup height. The linear relationship is to be expected as a greater amount of material available for forming should logically result in a taller cup. This is illustrated more clearly in Figure 5.25. The amount of material, which undergoes deformation during forming, is equal to the cut edge area minus the cup base area. The punch diameter and nose radius governs the dimensions of the base of the cup. Therefore, it is a donut shape (shaded in Figure 5.25) that becomes the material in a drawn cup wall. It is expected that extrapolation of the cut edge data in Figure 5.20, back to the x-intercept at zero cup height should be exactly that of the punch diameter, 66.79mm. However, this is not the case. An increase of 7.5% to approximately 72mm is shown experimentally.

Further consideration of this highlights two notable points. Firstly, the punch nose would be expected to influence the x-intercept in a positive direction. A disc drawn

over the punch nose has some curvature in the shoulder of the inverted cup. For this punch, with a nose radius (P_{rad}) of 1.2mm, a certain amount of material is required before a vertical wall is achieved. Assuming perfect punch nose profile, which equates to a quarter the radius circumference for a right-angle change in cup to base wall profile, then the material required in a cup shoulder (C_{sh}) is defined as:

$$C_{sh} = \frac{1}{4}.2\pi(P_{rad}) = \frac{\pi}{2}.(P_{rad}) \qquad \textbf{Equation 23}$$

$$C_{sh} = \frac{1}{4}.2\pi(1.2)$$

$$C_{sh} = 1.88\text{mm}$$

These calculations show that 1.88mm of material is required to produce the first 1.2mm in cup height. This would then cause a vertical shift in the whole data set. However, it is unlikely that this would account for the whole of the experimentally measured difference of the intercept value from Figure 5.20. The second question to consider is how can a disc with a diameter greater than the punch diameter have zero cup height? In extrapolating the data back to zero cup height, it is assumed that all the cups are formed in the same way and have the same physical properties. Although all the cups are produced in the same manner, their wall profiles differ with cut edge dimensions. The 100mm diameter disc exhibits pure draw because the wall thickness is always equal to or greater than its original gauge. The 120 and 125mm Discs exhibit a degree of thinning in the lower regions of the cup wall with the largest cut edge dimension. It is therefore acceptable to assume that stretching in the cup wall at larger cut edge dimensions skews the best-fit line for the data.

The final point of discussion for this topic must consider the inability to produce a successful cup at a cut edge diameter of 135mm. This experiment gives a draw ratio (Equation 24) of 2.02.

$$D_{rat} = \frac{C_{edge}}{P_{dia}} \qquad \textbf{Equation 24}$$

$$D_{rat} = \frac{C_{edge}}{P_{dia}} = \frac{135}{66.79} = 2.02$$

No exact data exists to calculate the limiting draw ratio, LD_{rat}, for film laminate. However, literature suggests that LD_{rat} for lacquered packaging grade steels is in the region of 2 to 2.2. Therefore, it is understandable that the 135mm cut edge diameter disc did not form successfully under a draw ratio of 2.02. It is also possible to assume that the limiting draw ratio for this particular film laminated material is in the region of 2.0.

Punch diameter

The effect of punch diameter on the drawing characteristics is closely related to the cut edge dimensions. As shown in literature and Equation 24, the draw ratio is a governing factor in the amount of material available for forming in a given operation. Figure 5.25 shows a donut shape of material that must be drawn into the cup wall, with the central circle becoming the cup base. Therefore, if the punch diameter (P_{dia}) is increased for a given cut edge, cup height must decrease. It is this relationship, which is shown in Figure 5.22. Three punch diameters are shown as a plot of cup height against cut edge. In all cases larger punch diameters generate a shorter cup for a given cut edge size. Additionally, increasing the cut edge diameter increases the amount of material available for drawing into the cup wall. It was shown in the previous subsection that the change in cup height due to cut edge is a linear relationship, therefore, extrapolation of the data back to zero cup height should reveal a value approximate to P_{dia}. This is indeed the case, both the 88.9 and 90.42mm diameter dies show better closer correlation to the actual zero value than the 66.79mm punch. This could be for a number of reasons. Firstly, the larger punch sizes are expected to experience less wall thinning than the smaller punch sizes because of the lower draw ratio. It was shown in the previous subsection that larger cut edge sizes (and hence draw ratio) caused a greater amount of lower cup wall thinning than cups produced at smaller draw ratios. It can therefore be assumed that the 88.9 and 90.42mm punches do not result in significant amounts of wall thinning when using cut edge diameters of 120 and 125mm. This would result in less distortion of the best fit line for these data sets, resulting in a more accurate zero cup height extrapolation in Figure 5.20.

5.2.4 Effect of lubrication

Lubrication weight

Figure 5.23 shows how cup height changes with lubrication weight. At very low levels the cup height is tallest, with a 40.08mm cup produced at a lubricant weight of 0.002g. In the lubrication weight range from 0.002 to 0.01g per disc, cup height decreases by over 1.5mm. This is to be expected, addition of a lubricant to a surface change the frictional characteristics, facilitating material flow. A state of diminishing returns becomes apparent, for every increase in lubrication level the change in cup height is minimised. Between 0.01 and 0.03g per disc, cup height decreases by a further 0.7mm. From 0.03 to 0.05g per disc change in cup height is minimal, only a 0.2mm decrease is detected. At these highest lubrication levels it can be argued that the excess of lubrication effectively causes the blank holder load to be reduced, due to decreased resistance to movement between the sample and tooling. It is also notable that the spread in data is largest at lower lubrication levels. Some failures were produced in this range. These results suggest that, to maximise cup height, lubrication levels should be kept as low as possible. However, enough lubrication is required to eliminate failures and produce consistent, repeatable results.

Lubricant type

A range of commercial lubricants was compared to the oil/alcohol emulsion used throughout this document. A petrolatum wax, Valspar and EZ11 were trialled. Figure 5.24 shows how the resultant average minimum cups for the experiment. Interestingly, the control gave the tallest cup in these experiments of 40.0mm, whilst one of the most popular commercial lubricants (valspar) gave the shortest (38.89mm). The results prove that there is a variation in forming characteristics of film laminate materials caused by lubrication type. Additionally, doubt must be cast onto direct comparison of published findings without detailed knowledge of lubrication procedures. However, trends in data from identical experiments are still valid.

5.2.5 Prediction of cup height

Analysis of standard film laminated materials

To build an equation to accurately predict cup height we must make some assumptions. In the simplest case a disc of known cut edge (C_{edge}) is drawn into a cup by a punch with external diameter (P_{dia}). The die diameter is of a radius that does not cause any stretching, or wall thinning. The punch nose radius is square and does not cause any changes in wall profile. In this scenario the material is of unit thickness, which does not change when drawn. This results in a cup with uniform wall thickness equal to the original material gauge, i.e. equal volume. Therefore, with no material losses the circumference of the punch multiplied by the height of the cup (h), must equal the cut edge area minus the base area of the cup:

$$h.\pi.t.P_{dia} = \pi.\left(\frac{C_{edge}}{2}\right)^2.t - \pi.\left(\frac{P_{dia}}{2}\right)^2.t$$

$$h.P_{dia} = \frac{C_{edge}{}^2}{4} - \frac{P_{dia}{}^2}{4}$$

$$h = \frac{C_{edge}{}^2}{4.P_{dia}} - \frac{P_{dia}{}^2}{4.P_{dia}}$$

$$h = \frac{1}{4}\left[\frac{C_{edge}{}^2}{P_{dia}} - \frac{P_{dia}{}^2}{P_{dia}}\right]$$

$$h = \frac{P_{dia}}{4}\left[\frac{C_{edge}{}^2}{P_{dia}{}^2} - \frac{P_{dia}{}^2}{P_{dia}{}^2}\right]$$

$$h = \frac{P_{dia}}{4}\left[\left(\frac{C_{edge}}{P_{dia}}\right)^2 - 1\right] \qquad \textbf{Equation 25}$$

By substitution of the $\left(\frac{C_{edge}}{P_{dia}}\right)$ factor within Equation 25, by D_{rat}, the draw ratio (defined previously in Equation 24), we can simplify to Panknin's prediction of cup height, which is shown in Equation 26.[76]

$$h = \frac{P_{dia}}{4}\left[D_{rat}{}^2 - 1\right] \qquad \textbf{Equation 26}$$

This equation shows reasonable correlation with results obtained experimentally. Taking the data highlighted in Figure 5.20 and plotting this as Figure 5.26 with the predicted height from Equation 26, we see an R^2 value (for y = x) of approximately 92%. However, due to the assumptions made in derivation of this equation no account is taken of planar anisotropy visible in the form of earing on the cup perimeter. This helps explain why all data is lower than predicted. Minimal earing is very unlikely in double reduced material due to the high percentage total reductions employed during manufacture of this type of substrate. Therefore, Equation 26 predicts a value midway between the average minimum and maximum cup height values. Indeed, averaging of these two measurable data sets gives a cup height of 40.34mm, much closer to the predicted value; only 2.9% difference. However, all DR film laminated materials do exhibit earing. Additionally, it is the minimum cup height that is of importance to can makers because it governs the amount of wastage lost through trimming. Therefore, a more accurate prediction of minimum cup height, which includes some of the discrepancies highlighted from Equation 26 for laminated materials needs to be found.

Lyons and Hudd,[101] have analysed studies of earing in aluminium alloys conducted by several other authors. This was then adapted for steels used for drawing applications and backed up by experimental results. The gauge range used in these studies was 0.8 to 1.2mm. They concluded that the following equation to predict earing in steels shows linear correlation with actual cup height values and that it would also be valid for packaging grade steels.

$$\Delta h = m\left(\frac{Cedge^2 - Pdia^2}{Pdia}\right)\left(\frac{Cedge - Pdia}{Pdia}\right)^{1.5} \qquad \textbf{Equation 27}$$

where m is a dimensionless value, which varies between steels.

The above equation is used to predict the magnitude of earing (Δh), the difference between average minimum and maximum cup heights. If we now calculate m for substrate in material no.12 in Table 4.1, it is found to equal approximately 33. Use of this value in Equation 27 for three different sets of cut edge disc size at a punch diameter of 66.79mm is shown against actual values in Figure 5.27. This shows measured earing against predicted values of earing or cup height by Hudd and Lyons or Panknin. It is clearly seen that over the range of data investigated, Panknin's

prediction of cup height (Equation 26) shows excellent correlation with measured earing. Therefore, Equation 26 is equal to the best fit line shown in Figure 5.27, shown below in Equation 28.

$$h = \frac{Pdia}{4}\left[\left(\frac{Cedge}{Pdia}\right)^2 - 1\right] = 7.64\Delta h + 13.33 \qquad \textbf{Equation 28}$$

This equation shows that earing is proportional to some value, which for this material equals 7.64 plus a constant, in this case 13.33. The constant suggests that at zero earing we still have some cup height, i.e. at a cup height of 13.33mm, $\Delta h \cong 0$. This is very likely. As cup height decreases, earing decreases, to point where it is not accurately measurable. Therefore, addition of this constant to Equation 28 is acceptable.

Rearranging Equation 28 for earing Δh, the following is derived:

$$\Delta h = \frac{1}{7.64}\frac{Pdia}{4}\left[\left(\frac{Cedge}{Pdia}\right)^2 - 1\right] - \frac{13.33}{7.64}$$

$$\Delta h = \frac{1}{n}\frac{Pdia}{4}\left[\left(\frac{Cedge}{Pdia}\right)^2 - 1\right] - \frac{c}{n} \qquad \textbf{Equation 29}$$

There are two new factors in Equation 29, n and c. n is a material constant, which for the laminate under investigation equals 7.64. Lyons and Hudd also found a need for the addition of a material constant to their earing height equation. Since not all materials have equal magnitudes of earing, this is a valid assumption. n is expected to change with factors such as steel chemical composition and percentage total reduction of the final product from the original hot band gauge. c is the intercept value for cup height at zero earing, which for this data equals 13.33. This concept is explained above.

To predict cup height we must combine a few of the previous equations. We have stated in the assumptions for Equation 26 that no earing is allowed for. Therefore this prediction is too high and we must take into account half of the earing value, i.e. only half of Equation 29 need be removed to calculate minimum values. This equation then becomes:

$$h = \frac{P_{dia}}{4}\left[D_{rat}{}^2 - 1\right] - \frac{1}{2}\frac{P_{dia}}{4n}\left[D_{rat}{}^2 - 1\right] - \frac{c}{n} \qquad \textbf{Equation 30}$$

However, Equation 30 does still not take into account the material 'gained' over the punch nose radius, as discussed in section **5.2.3**. It was stated in the assumptions of Equation 26 that the equation deemed the punch nose to be square, this is not the case. For determination of the material used in the cup shoulder profile we must take into account the material in contact with the punch nose radius. This is indicated more clearly below.

$$Gained\ height = 2P_{rad} - \frac{\pi P_{rad}}{2}$$

$$Gained\ height = P_{rad}\left[2 - \frac{\pi}{2}\right]$$

Therefore, addition of this value from the previously developed equation is also required to provide a meaningful predictive cup height equation.

$$h = \frac{P_{dia}}{4}\left[D_{rat}{}^2 - 1\right] - \frac{P_{dia}}{8n}\left[D_{rat}{}^2 - 1\right] - \frac{c}{n} + P_{rad}\left[2 - \frac{\pi}{2}\right] \qquad \textbf{Equation 31}$$

A plot of the predicted values from Equation 31 against actual cup height values for a range of different cut edge sizes and punch diameter of 66.79mm shown in Figure 5.28. This shows an R^2 correlation of 0.998 for the data.

Equation 31 can now be used to predict cup height for a range of punch and cut edge sizes. The other two punch diameters used in this document were 88.90 and 90.42mm, with successful cups produced from a cut edge of 120, 125 or 135mm. A plot of all these data points for measured against predicted minimum cup height is shown in Figure 5.29. Although good correlation is shown for the results, the larger punch sizes are all grouped below the y = x line. There are only two places where this anomaly could have occurred. The first is in the assumption that the variable c is constant for all punch sizes. Further investigation of this value is therefore required. However, this has proved difficult. Of the four cut edge dimensions investigated 100, 120, 125 and 135mm, only the largest three could produce measurable cups over 88.9 and 90.42mm punch diameters. Although minimum cup height showed good consistency in recorded values, the earing value was variable. Therefore, it has proved difficult to further analyse this data. A wider range of punch diameters or cut edge sizes would be

required to fully analyse the assumption that the c constant is variable with either punch size or draw ratio.

The second area where discrepancies are likely between the developed predictive equation and reality is the assumption that wall profile remains uniform. Data in Figures 5.13 & 5.21 clearly show that all examined cups have a non-uniform profile. All wall profiles show that the top of the cup is thicker than at the base. It has also been suggested (section **5.2.3**) that higher draw ratios cause lower-wall thinning. None of these factors are considered in the above predictive equation. To improve the accuracy of the prediction, cup wall profile variations would need to be included. The simplest method would be to assume top wall thickening is proportional to cup height, i.e. the profile represents a wedge. However, this approach can have the same overall volume as the constant wall thickness approach in Equation 31, assuming that the gauge midway up the wall equated to the nominal laminate thickness. Experimentally, this has been shown to do approximately correct, with the 120 and 125mm cut edge cups (Figure 5.21) equal the nominal gauge at 40% and 54% of wall height. This theory explains why the prediction derived in Equation 31 remains accurate despite its simplicity. In reality the wall profile does not represent a wedge, but part of a cosine curve. More mathematical development would be required to assess this relationship over a larger number of variables, thus warranting further analysis of this avenue not viable using the current data streams.

Quantification of material characteristics

We have clearly shown that elements in each of the above subsections (substrate properties, polymer coating and tooling issues) influence forming characteristics. Therefore, to achieve a more accurate prediction of cup height we must develop a method of prediction, which takes the most significant factors into account.

The factors, which had greatest influence on cup height within the experiments conducted in this document, were substrate thickness, strength and die radius. The first of these factors, substrate thickness was shown to be a linear relationship across the data range investigated 0.14 to 0.26mm. Within this range the equation for cup height across both die radii investigated is shown in Equation 32:

$$h = (S_{th}.16.51) + c \qquad \textbf{Equation 32}$$

The constant c, is dependent on die radius. In these experiments c equals 37.6, for the 1.0mm die radius and 39.35 for the 2.0mm die radius. To incorporate Equation 32 into the predictive equation discussed previously, substrate thickness would have to be taken relative to the 0.18mm gauge used in those experiments.

The second variable, which has a marked effect upon drawn cup height, is substrate strength. This was found to be a non-linear relationship between the investigated range of DR520 and DR620 nominal strength substrate. (N.B. Actual ultimate tensile strength readings were on average 30MPa lower than the nominal values.) However, for the 2.0mm data set a linear equation best suits the data. However, this equation would not be as accurate for cups produced over the 1.0mm die radius. The equation that best describes the experimental data is shown below in Equation 33. Again, a modulus value from 580MPa, used within the previous experiments, would be required to compare directly with Equation 31.

$$h = \frac{S_{st}}{116} + 34.69 \qquad \textbf{Equation 33}$$

The final variable, which was found to have a significant effect upon cup height, was die radius. A non-linear relationship was also found to be evident, with cups unable to be produced at a die radius of 0.75mm for materials 3 and 6 in Table 4.1. For the 0.18mm DR580 material an approximate guide to the change in up height with die radius is shown in Equation 34. However, further investigation would be required to assess the accuracy of this suggestion.

$$h = -2.34 \ln(Drad) + 39.66 \qquad \textbf{Equation 34}$$

The other factors considered within this document have minimal influence on drawn cup dimensions. The majority of the polymer coating related issues; pigmentation, type and thickness were found to cause differences in cup height less than the spread in the data. It is therefore acceptable to state that these factors can be assumed to be negligible in comparison to those previously considered in development of a predictive cup height equation.

6 Biaxial Stressing of Film Laminated Materials

In this study biaxial stressing occurs primarily during dome production, or simulation, in can making. Because the can base, or disc, allows only limited amount of drawing to occur, stretching is evident during forming. Therefore, a biaxial stress field is apparent in all doming operations.

It has been shown that certain film laminated materials exhibit an eliptical craze whitened area upon application of a biaxial stress. This section of work investigates this phenomenon in more detail.

6.1 Results

As described earlier, in section **4.2.3**, there were problems assessing craze shape or dimensions, which vary over a very small area within the crazed regions. It was decided to assess crazing performance by measurement of the affected area. The dome simulation usually produces crazing visible as an ellipse, or elliptic ring, around the central region of the specimen. It should also be noted that, after deformation of the sample, all crazing (when present) takes approximately two hours to fully appear. After this time no further change takes place.

6.1.1 Substrate properties

Tensile data for the three investigated substrates is shown in Tables 4.4, 5.12 and 5.13. The fully aged or 'old' laminate, 0.17mm DR620 (no. 1 in Table 4.6) shows an average UTS and proof strength of 577.0 and 571.3 MPa longitudinal and 661.0 and 652.1 MPa transverse to the rolling direction. The ratio of strengths perpendicular and parallel to the rolling direction is approximately 1:1.15. For the recently produced or 'fresh' laminate, 0.17mm DR620 (no. 2) the UTS and proof strength average at 589.4 and 586.7 MPa. The blackplate based laminate (no. 3) averages are 566.4 and 563.3 MPa respectively. In all cases variation in UTS along and across the tested area is less than 30.0 MPa.

6.1.2 Ageing of fresh plate

Fresh laminate material was produced on the Ferrolite line, using a 0.17mm ECCS DR620 CA material with 15μm clear PET on both sides. The material was produced at three different preheat temperature regimes, 165°, 215° and 230°C, providing three sample groups for analysis. Discs from each group were then formed into domes at various time intervals following lamination and the resultant craze appearances were inspected and indicated in Figure 6.1. The results show that crazing is not visible in any form when a biaxial stress is applied in the first few days following production of the fresh laminate. However, within one week some crazing is evident at all three preheat variables. The shape of the curve shown by the data is almost identical for the three temperature regimes. Analysis of the difference in the size of the crazed area between the three sample groups gave a maximum value of 0.125×10^{-3} m^2 at two days after production. The crazing reaches a limiting value of 3.4×10^{-3} m^2, approximately 10 days after production. After this time no subsequent change in the affected area is noted.

Figures 6.2 show Differential Scanning Calorimetry (DSC) analysis of the clear coating of a typical film laminated material at various time periods after lamination. Line 1 is representative of a material 2 days after production, line 2 is of the same batch of material 100 days after lamination. Line 3 shows the same material as in line 2, but it was heated to a temperature of 95°C and rapidly cooled to room temperature prior to testing. The area of interest in all three DSC traces is around the T_g, glass transition temperature (~75°C for PET). The height and area of the T_g peak clearly increases with time period after production. Line 3 also shows that heating of the sample to an elevated temperature and rapidly cooling prior to assessment greatly reduces the magnitude of the glass transition peak.

6.1.3 Prestoving experiments

Discs of material no. 1 in Table 4.6 were heated or stoved to various temperatures between 60° and 100°C for one minute. At time periods from 60seconds to 3 months following stoving, samples from each temperature regime were deformed using the standard dome simulation testing procedure. Figure 6.3 shows how the area of crazed

polymer coating varies with prestoving temperature. A control (unheated) sample is also shown for reference. It is clearly seen that with prestoving temperatures of ≤ 70°C, no difference is discernible when compared to the control. The crazing magnitude at these temperatures is effectively constant at $3.4x10^{-3}$ m^2 at all periods following stoving. This is equal to the limiting value obtained for fresh plate. Samples heated to temperatures between 75° and 90°C show an elimination of crazing for certain time periods after stoving. Within this range, the time period after which the dome simulation results in crazing differs. At 75°C, crazing is visible following the application of a biaxial stress at a time period of 120 seconds. The onset of crazing is delayed with increasing prestoving temperatures to a maximum of 3 days at 90°C, i.e. no crazing occurred on a sample heated to 90°C, providing it was deformed within 3 days of prestoving. However, in all cases crazing occurs on all samples, after deformation, at time periods after prestoving of greater than one week. At prestoving temperatures ≥ 90°C the time period after heating, at which crazing is detected following deformation is constant at three days. At these higher prestoving temperatures the total crazed area goes from zero, when the sample is deformed 1 day following stoving, to easily detectable, 3 days following stoving, with no intermediary levels detected. All prestoving temperature investigated exhibit crazing, upon deformation, at three months after heating, with identical magnitudes of $3.4x10^{-3}$ m^2

6.1.4 Lubricant additions

Rolling lubricants

A comparison of the standard laminate material (no 2 in Table 4.6) with the blackplate material (no.3) is shown in Figure 6.4. The craze susceptibility is shown as total crazed area against time after production. Neither material exhibits crazing, when deformed in the first few days after production. After one week the standard material shows crazing. This area increases to approximately $3.1x10^{-3}$ m^2 at 16 days after lamination, but does not become any more severe after longer times. Conversely, the laminate material made using the blackplate manufacturing route did not exhibit crazing at any time after production.

Lubrication applied prior to forming

Four different lubricants were applied to the surface of a laminate material and deformed at various time periods after addition. The lubricants in question were a petrolatum wax, Dioxylsebacate (DOS), Acetyltributylcitrate (ATBC) and Butylstearate oil (BSO). The lubricants were applied to the sample groups of materials 1 and 2 in Table 4.6; representing 'old' (or fully aged) and 'fresh' (recently laminated) material. Figures 6.5 and 6.6 show how the craze susceptibility changes over time for the two laminates in question. In each case an unlubricated control is also present in each figure for reference.

Figure 6.5 shows how the addition of the lubricants to the 'fresh' laminate material influences the craze susceptibility with time. At all short periods after application (≤10 days) the wax showed no or less crazing than the control. At one month after production (and hence lubricant addition) the petrolatum wax shows a crazed area of $3.5x10^{-3}$ m^2, a difference of less than 2.5% compared to the control. The rolling lubricants investigated (DOS, ATBC, BSO) show craze susceptibility to be increased in relation to the control. At one day after production DOS, ATBC, BSO and the control showed crazed areas of $3.4x10^{-3}$, $3.6x10^{-3}$, $5.0x10^{-3}$ and 0.0 m^2, respectively. By one month these values had increased to $5.0x10^{-3}$, $8.6x10^{-3}$, $9.2x10^{-3}$ and $3.4x10^{-3}$ m^2. This represents a respective increase in severity of 47, 153 and 171% for the three rolling lubricants in comparison to the control at 28 days.

A similar data series is shown in Figure 6.6 for the 'old' laminate material. N.B. Lubricants were applied to the surface of each specimen approximately six months after production. In this case the x-axis labelling shows time period test conducted after lubrication and not laminate production as shown in the previous figure. Of the four lubricants tested, two trends emerge. The petrolatum wax does not change the crazing area in relation to the control. At all time periods after lubrication the affected area remains constant at approximately $3.5x10^{-3}$ m^2. This differs from the previous experiment where the addition of the wax reduced the severity of crazing at early time periods after application. The second trend from Figure 6.6 is in relation to the rolling oils. At all time periods after lubrication the crazed area is larger than the control. DOS, ATBC, BSO and the control show a crazed area of $8.1x10^{-3}$, $8.5x10^{-3}$, $9.3x10^{-3}$

and $3.4x10^{-3}$ m^2, four weeks after addition of the lubricant. This represents an average increase of 150% in crazed area for the rolling lubricants, compared to the unlubricated samples. The relative severity of each lubricant is identical in the information shown in Figures 6.5 and 6.6.

6.2 Discussion

It has been noted that the internal PET clear coating on some domed containers had a tendency to form a series of circumferential white rings around the deformed region. This section of work investigates the factors which affect the formation of crazes and highlights ways of minimising or eliminating the problem.

6.2.1 Substrate properties

The tensile properties for all three materials investigated in this section are shown in Tables 4.4, 5.12 and 5.13.The average UTS and proof strength values for each material are within the acceptable range, as shown in Table 5.3. The range of tensile values taken parallel to the rolling direction show a variation of less than 30MPa for all three materials. Table 4.4 also shows the 0.17mm DR620 substrate has a ratio of transverse and parallel UTS values of 1:1.15.

The major axis ratio of elliptical crazing rings (Table 4.7) is the same as that shown in Table 4.4, relating to longitudinal and transverse UTS properties. The change in tensile properties transverse and longitudinal to the rolling direction is the most likely explanation why the crazes are formed in an elliptical pattern and not a circular one as would be expected from round-nosed tooling. In all cases crazing appears to follow the line of maximum strain distribution.

6.2.2 Ageing of film laminated material

As explained in the literature survey, section **2.4.2**, amorphous polymers have a tendency to age. In this case, although PET is a semi-crystalline polymer, when it is rapidly cooled from above its crystalline melting point, as in the Ferrolite production process, a non-crystalline amorphous structure is produced. Amorphous solids are not in thermodynamic equilibrium.[102] The excess volume trapped between molecules is referred to as free volume. A system that is not in thermodynamic equilibrium will attempt to lower its energy state over time, primarily by continued packing of the structure. The changes in the structure of a polymer and it's effect on mechanical properties is known as free volume relaxation, enthalpy relaxation or physical

ageing.[52] A full explanation of the free volume concept is shown in the literature survey.

Film laminate materials also show signs of ageing. The DSC plots in Figure 6.2 show an increase in the area under the glass transition peak with time. This suggests that alignment of the polymer structure is taking place over the investigated period. There is evidence to show that the glass transition temperature increases with the amount of packing in a semi-crystalline polymer.[99] This ageing phenomenon may also explain why crazing does not occur when a dome is formed immediately after production. It is possible that the polymer has to age a certain amount before craze formation begins. This indicates an interaction between ageing and crazing in film laminate materials. Theory suggests that at a short ageing time there is free volume in the polymer structure. This results in a lower yield stress and easier global plastic flow, which reduces the likelihood of localised deformation observed as crazing. A number of studies have shown ageing time increase brittleness.[103, 104, 105] However, its effect on environmental stress cracking is not conclusive.[54]

Analysis of newly produced 'fresh' film laminate material is assessed in Figure 6.1. It is shown that for the first few days after lamination no crazing is evident under the application of a biaxial stress. However, within one week of the date of lamination crazing is clearly visible under the same stress conditions. These results do not vary with Ferrolite line preheat temperatures during production.

The occurrence of crazing can be attributed to either one of two theories. The first is a standard nucleation and growth process, which causes most thermoplastics to craze under sufficient stress. The second is a form of environmental stress cracking (ESC), whereby an external environmental factor combined with an applied stress causes failure at a point lower than would normally be expected if either the stress or the environment were eliminated. The visible damage associated with this mechanism can be observed as macroscopic crazing, also seen as local whitening in the region of maximum stress. This has resulted in an additional term environmental stress crazing.

6.2.3 Prestoving of laminated material

The results in Figure 6.3 show that when a sample of 'old', and therefore craze susceptible, film laminated material is heated to a temperature in excess of the glass transition temperature (Tg) of the polymer, ~75°C for PET, crazing is removed for a period of time. Eventually crazing reoccurs during, or a short while after, the application of a biaxial stress. The time at which crazing first reoccurs after stoving increases with increasing stoving temperature. At 75°C it takes less than two minutes for crazing to reoccur, whilst at stoving temperatures above 100°C it takes up to 3 days before the application of a biaxial stress results in crazing. The results appear to show a limiting value of maximum crazing delay being reached at temperatures above 90°C; this is more evident in Figure 6.7, where time to first craze reoccurrence after stoving is plotting against stoving temperature. However, with only two sets of data above 90°C it is difficult to conclude if this is an absolute limiting value or whether it would increase further with higher stoving temperatures. One other point of note, clearly evident from Figure 6.3, is that crazing in the higher prestoving temperature data sets jumps from no detectable crazed area to a severe sample between one and three days. This is most likely explained by the large time differences between assessment at longer time intervals after stoving. For the 75°C and 80°C stoving temperature data, it takes between one and three days to reach a detectable crazing level. If it is assumed that the duration required to reach detectable crazing is constant at all stoving values and the only result that alters in each data set is the initial delay to first craze occurrence, then it is feasible that smaller crazed areas could pass by undetected between the 1 day and 3 day assessment times in the 90°C and 100°C data sets. This is backed up by the 80°C data set where crazing increases from very low levels to an easily detectable area within six hours. The data in Figure 6.7 also showed that provided a laminate material, of any age, was stoved to a temperature above the polymer's glass transition temperature immediately prior to forming the dome then crazing would not occur.

6.2.4 Lubricant additions

Rolling lubricants

Figures 6.5 and 6.6 show that the addition of excess rolling lubricants to the surface of the laminate increases the severity and area of crazing. This leads to the likelihood that the mechanism for craze formation in laminated materials is a form of environmental stress cracking. This idea is upheld when we compare a similar specification laminate, which has not gone through the electro-chromium coating lines. This blackplate production route had not had any rolling oil applied to the surface of the substrate prior to film application and does not craze at any time period after production. Further evidence of this mechanism is shown in Figure 6.3, where no crazing occurs within the first few days of lamination. Literature states that PET is susceptible to ESC in many applications other than for film laminated material.[51] Therefore, by definition of environmental stress cracking, removal of the stress or the active medium would eliminate crazing. However, there is still the possibility of an ageing / crazing interaction affecting results. Methods of achieving film laminate materials that do not craze are more fully discussed in the prevention section of this report.

Lubrication applied prior to deformation

Results in Figure 6.5 have shown that the application of a petrolatum type wax to the surface of the laminate immediately after production delays the onset of severe crazing by up to four weeks. There are two possibilities as to why this occurs. The first is that the wax forms a simple barrier layer preventing any interaction between the polymer and the external environment. The second is the wax is absorbed into the to upper layer of the polymer, causing a local expansion of the surface, preventing the critical craze initiation stress being reached. If the correct mechanism for the craze formation is one of ESC then the problem environment is already present at the interface between the polymer and the substrate. This points towards the likelihood that the wax is preventing initiation of the crazing by expansion of upper polymer layer, thus reducing the stress levels present. However, it is also possible that plasticisation and blunting of the stress concentration occurs. Published data suggests that compressive stresses caused by the wax is the more likely scenario.[106, 107] The

wax will evaporate over time, explaining why the crazing occurs at a later time period when these properties are reduced to a level where crazing cannot be prevented. This assumption agrees with data in Figure 6.6, which shows no difference in crazed area between the petrolatum based wax and a control sample, lubricated six months after lamination. The activating medium for ESC has already diffused into the coating and cannot be delayed by application of a wax to the surface. This also suggests the wax must be applied before sufficient ageing has occurred to help delay crazing.

All rolling oil lubricants (DOS, ATBC and BSO) when applied to the surface of a laminate material increase the severity of crazing. This is true for both aged and recently produced laminate materials and is shown in Figures 6.5 and 6.6. Clearly samples treated with these lubricant exhibit far greater crazed area than an unlubricated control. It is therefore quite possible that these lubricants, when present at the interface layer between the substrate and polymer, have a detrimental effect on craze susceptibility of that plate. Although these oils are present in much smaller quantities on a commercial processing route than applied in these experiments the fact that they accelerate the crazed area helps sustain the idea that craze growth in PET coated film laminates is based on an environmental stress cracking (or crazing) mechanism.

The solubility parameters for each of the ESC inducing lubricants has been calculated from published information on individual solubility effects.[108] A summary of this data is shown in Table 6.1. The total solubility values for PET, BSO, ATBC and DOS are 20.5, 17.7, 19.0 and 18.0, respectively. Theory suggests that ESC is more likely when the active liquid, or lubricant, is close to that of the polymer (PET); in this case ATBC would be most likely to induce ESC followed by DOS and BSO. This differs from the experimental data collected, which shows that BSO exhibits the greatest degree of crazing followed by ATBC and DOS. Since the total solubility parameters are very close for the investigated liquids and polymer other effects such as molecular weight and diffusion rates influence the results and the prediction becomes less accurate. Although the experimental data differs from the theoretical order of predicted ESC attack, it does show that using solubility parameters is a good way to highlight potentially dangerous liquid polymer combinations and has use as a robust rule of thumb for this type of application.

6.2.5 Prevention

There are two ways to commercially combat the craze phenomenon as highlighted in this document. The best solution would be to eliminate the problem by changing the process route. However, if this is not feasible then temporary removal or delay of crazing might provide a workable, short-term solution.

It seems apparent from the evidence delineated above that the mechanism encountered to produce crazing is one of environmental stress cracking. Therefore, removal of the stress or the active medium would eliminate the problem. Since the dome of the can is an integral part of the design, used to withhold pressure produced during sterilisation or filling, application of a biaxial stress is unavoidable. Hence, the second alternative must be examined. Removal of the rolling lubricant that is applied to the sheet prior to lamination has shown to negate the crazing problem; thus providing the obvious solution. However, the rolling oils are applied to the strip for lubrication reasons for other commercial applications. Removal of the oils would be an acceptable solution for film laminated products. However, in the event of a coil being rejected at the laminating line, would not allow alternate rerouting of that coil to occur due to the necessity of the rolling oils for all other applications. The secondary alternative would be to change the type of rolling oil in use. However, all three of the most common oils (DOS, ATBC and BSO) have been shown to accelerate the crazing problem. The number of alternative oils is also limited due to their necessity to be FDA and Kosher approved.

If removal of the rolling oils is not commercially viable, then an acceptable alternative must be found. The first solution would to delay the onset of crazing to a time period after the dome has been formed, thus avoiding the problem. It has been shown that the application of a petrolatum type wax to the surface of the laminate material directly after production delays the onset of crazing by up to four weeks. For plate older than four weeks a second solution must be identified. Heating the 'old' plate to a temperature above the glass transition point of the polymer, namely 75°C for PET, allows relaxation of the stresses to occur. It has been shown that if the plate is heated to 100°C for 60 seconds, then crazing will not appear during the application of a

biaxial stress for up to three days. If the stress is applied at a time period after three days, then crazing will once again become evident. Both of these solutions provide a short-term solution to removal of the crazing phenomenon using the current production route, involving the application of rolling oils.

However, if the substrate route for film laminate materials, which uses non-passivated substrate was fully developed a solution to the problem could be achieved. This type of material avoids the passivation lines and hence has no rolling oil applied. Material manufactured in this way did not craze at any time period after lamination. Therefore, this production route would not only have the obvious cost advantages of missing out the expensive coating stages, but would also not be susceptible to crazing in film laminated form.

7 Summary Discussion

It has been shown in the previous two sections of this document that the forming of film laminate materials is a complex subject, with many factors that influence the integrity and drawn height of a container. Of the variables investigated in relation to single stage drawing of film laminate, some have proved more critical than others.

7.1 Drawing of film laminated materials

7.1.1 Substrate properties

Three areas relating to substrate properties were considered; passivation/coating, thickness and substrate strength. Of these, thickness had the greatest effect on drawn cup height. For a change in gauge from 0.15 to 0.26mm, cup height linearly increased by nearly 5%. However, this compares to an increase in material volume of 73%. This suggests that continual downgauging, which is a permanent pressure in can making will only have a marginal affect on drawn cup height.

The influence of base metal ultimate tensile strength has also been shown to have only minimal effect on drawn cup height. For analysis of materials of nominal strength 520 to 620MPa, an increase in container dimensions of 3.5% was noted. This suggests that both the above factors (strength and gauge) should be chosen in combination to achieve the required final can axial crush strength and dome reversal pressure and not to generate the required cup height.

The final area investigated, loosely related to substrate properties, is passivation or coating type. Comparison of an uncoated material, lacquered ECCS and a typical film laminate specification was conducted. It was shown that the uncoated material, when successfully drawn, generated a taller cup than the two coated alternatives. However, the uncoated material failed during drawing on over 50% of the samples, whereas the two coated alternatives only showed failure rates of 2.5% (film laminate) and 4% (lacquered ECCS). The film laminate material was less likely to fail at high blank holder forces than the lacquered ECCS. Further analysis of these two sample groups

showed less than 1% difference in cup height. For applications where film laminated materials compete directly with lacquer sheet, such as two-piece food cans, this shows that the former is not detrimental to container performance. Indeed, it could be argued from these results that film laminated feedstock is more tolerant of difficult forming procedures than lacquered ECCS.

7.1.2 Polymer coating

Four issues relating to the polymer coating in film laminate materials were investigated; type, thickness, pigmentation and laminating conditions. All factors were shown to have minimal effects on the drawn cup dimensions. The most surprising of these was pigmentation. A white pigmented film was found to exhibit identical results when compared to a clear coating on the same substrate. The titanium dioxide pigmentation particles were expected to interact with the tooling during forming. However, surface analysis of the coating showed no pigmentation particles protrude from the laminate film. This explains why no difference was found in drawn cup height. It also suggests that any other pigmentation colour that may be commercially developed in the future would be unlikely to detrimentally affect deep drawing characteristics.

It was also shown that laminating conditions do not alter the forming characteristics of polymer coated steels. However, whether adhesional properties were detrimentally affected was not considered. Concerns that laminating temperature regime influences forming characteristics has been unfounded and may allow further development of the commercial film application process.

Both polymer type and thickness were found to have little or no effect on drawn cup heights. This is probably due to the minimisation of any frictional differences under application of the large blank holder forces, employed during production of a cup. The results point to a likelihood that any new variation of the present coating range (glycol substituted PET, isophthalate addition, nylon top-coats) would have minimal effect on deep drawn properties. This reasoning also applies for development of new coatings such as polyethylene naphthlate or novel coatings including holographic and reflective films.

7.1.3 Tooling issues

All mechanical, tooling and sample dimension issues were grouped into this third section. It has been clearly shown that the cut edge and punch diameters have the greatest effect on cup dimensions of all the factors investigated. For a 66.79mm punch, increasing cut edge from 100 to 125mm resulted in a change in average minimum cup height from 19.98 to 38.17mm, an increase of 91%. Considering only the material which can be turned into the can wall from the starting disc, i.e. the ring excluding the width of the punch diameter, a simple predictive equation can be derived:

$$h = \frac{P_{dia}}{4}\left[Drat^2 - 1\right]$$

where P_{dia} is the external punch diameter and D_{rat} is the draw ratio for that forming operation.

However, this equation does not take into account earing and material gained over the punch nose radius in the first few millimetres of the can wall, hence it over-predicts height in comparison to the actual values. Further analysis of these factors helped development of the following equation:

$$h = \frac{P_{dia}}{4}\left[Drat^2 - 1\right] - \frac{P_{dia}}{8n}\left[Drat^2 - 1\right] - \frac{c}{n} + \mathrm{Pr}\,ad\left[2 - \frac{\pi}{2}\right]$$

where c is a constant 13.33, n is a material value, which in this case equals 7.64 and P_{rad} is the punch nose diameter.

For a given specification laminate material, this equation is 95% accurate for a wide range of cut edge and punch diameters. This provides can makers and designers with a useful indication of the dimensions required to achieve a particular drawn cup height. To transfer this equation to all film laminate materials considered in this document, further evaluation was required.

It was found that all laminated materials could be successfully formed over die radii of 3.33 times that of the total laminate thickness. This is substantially lower than the typical value of 8-10 shown in literature. These tighter die radii had the added benefit of producing a taller cup by resultant lower wall thinning. Additionally, coating integrity was found to be undiminished for all the die radii investigated.

Increasing blank holder force from a value near the lower end of the operating window to one approaching the upper limit results in a cup height increase of up to 2.5%. This highlights a way of achieving a small additional increase in cup height without having to alter cut edge dimensions or tooling specifications.

7.2 Biaxial stressing of film laminated materials

The application of a biaxial stress field to certain film laminated materials, as in the production of a can dome, was found to cause a whitened elliptical ring around the region of maximum stress. This whitened region was caused by a series of crazes, the majority of which were shown not to extend through the total thickness of the polymer coating. The production of crazing was only visible in the clear PET coatings investigated. It was found that this phenomenon was governed by a few critical factors. Firstly, recently manufactured laminate material did not craze if it was deformed within three days of lamination. At time periods of three to seven days after production, biaxial stressing of samples resulted in visible crazing which was discernible. At time periods greater than one week after manufacture, deformation of a typical film laminated material was shown to cause a significant amount of crazing. This suggests that freshly produced film laminate material has a shelf life of seven days. After this time, biaxial stressing of samples will produce crazing.

In order to understand the phenomenon fully, it was necessary to establish a mechanism for appearance of crazing in film laminate materials. During production of feedstock for film laminated products, a thin film of rolling oil is applied to the surface substrate at the end of the passivation lines. Application of excess rolling oils to the surface of a clear PET coated sample, prior to biaxial stressing, was found to dramatically increase the crazed region by as much as 150%. This led to the theory

that craze propagation and subsequent growth was a form of environmental stress cracking. By definition, elimination of either the critical stress field or the active media (the rolling oil) would result in a non-crazed material. Since in can making the former is unavoidable, attention was turned to the latter. Production of a specialist film laminate material, which bypassed the passivation line and therefore had no rolling oil was initiated. Biaxial stressing of this particular material did not cause crazing at any time after manufacture. This supported the ideology that craze production in film laminated materials is dominated by an environmental stress cracking mechanism.

Removal of the rolling oils for standard commercially produced laminate material was found to be uneconomic. A coil that is rejected ahead of the laminating line could not be rerouted to another application without the presence of the troublesome oil. Therefore an alternative method of combating the problem was necessary. Since elimination of the stress or the active media is unworkable, other avenues were explored. It was found that prestoving of samples prior to deformation resulted in uncrazed samples. A critical temperature was discovered at 75°C, which is approaching the glass transition temperature of PET. Samples prestoving at temperatures greater than 75°C were found not to craze if immediately deformed. In fact, prestoving of 90°C for 1 minute delays the onset of crazing following deformation by up to 3 days. This gives a second window of opportunity for can makers to produce biaxially stressed can domes without the appearance of unsightly and detrimental crazes. Heating a sample above its glass transition temperature removes any ageing or chain alignment that occurs over time in amorphous polymers. It was therefore concluded crazing production mechanisms are of an environmental stress cracking nature, but the film laminated material also needed to be of a sufficient age to allow the mechanism to propagate. The exact interaction of these ideologies is still unclear.

7.3 Industrial relevance of the current programme

The deformation of any polymer coated steel is affected by many factors. All these need to be fully understood by manufacturers, can makers and designers in order to produce a product that is fit for purpose. This study has concentrated on two main areas of forming; deep drawing and biaxial stressing.

Research into drawing of a range of steel polymer laminates has highlighted some interesting areas. Firstly, no difference was found in drawn cup dimensions for a material with differing coating pigmentation on opposing sides of the laminate, regardless of which surface was in contact with the tooling during forming. Secondly, no change in coating integrity was found after forming in coating integrity for any of the variables investigated. This provides can makers who use steel polymer laminates with reassurance that swapping between white and clear coating specification, for a given application, will have no adverse effect on a container's height or integrity. This highlights an area of versatility and potential grade harmonisation for the future.

Cup height dimensions have been accurately predicted for a standard steel polymer laminate specification. This equation remains robust for a spectrum of cut edge and draw ratios. Additional research has developed a number of other equations that can be combined with the first to predict cup height for other substrate nominal specifications. This information provides designers and manufacturers with information on which steel polymer laminates would be required to achieve the necessary component height in a single draw operation. This will then ensure minimum overengineering and provide cost benefits to the customer.

It has also been shown that both die radius and blank holder force influence wall thickness and hence height of a cup. With the focus of the industry currently on the development of the stretch draw process, as in the RBS project, this research gives guidance on which draw ratios, die radii and blank holder force provide optimum component height without risking component failure through tear-off.

The second area of investigation within this study considered the biaxial stressing of film laminated materials and the tendency for certain variables to develop stress

whitening. This area of research is of most use to manufacturers as a quality control system. The investigation clearly shows that excessive rolling oils in combination with a clear polyethylene terephthalate coated laminate of a certain age results in environmental stress crazing of the product. This information has a number of uses; firstly when poor control is prevalent on rolling oil levels the substrate in question should not be scheduled into an application that will require a biaxial stress laminated product, as in dome formation or profiled components. Secondly, in the event of a product with potential to craze reaching the customer guidance can be given to allow prestoving of the material. This enables a possible area of coating weakness and potential customer claim or costly product recall to be avoided. Finally, this area of research highlights the need for a long term production route that does not apply rolling lubricants on substrate destined for steel polymer laminate applications. This could be achieved by running large volumes of material destined for polymer coated coating at a time and turning off the oiler. However, this avenue has a number of potential routing problems. Alternatively and perhaps more excitingly this investigation adds weight to the argument for development of a blackplate based laminate. This provides obvious benefits in both ease of routing through the plant and large savings by elimination of the costly chrome coating lines.

The issues highlighted in this document should allow everyone involved in production, design, and deformation of film laminated materials to manufacture containers to suit the needs of consumers of the 21st Century.

8 Conclusions

8.1 Drawing of film laminated materials

Effect of substrate properties

1. All substrates investigated within this document have shown consistent mechanical properties, well within acceptable limits.
2. Comparisons of blackplate, lacquered electro-chromium coated steel (ECCS) and film laminate materials showed the uncoated material produces the tallest cup. Little difference in height was noted in the coated material.
3. Film laminated materials exhibit more consistent cup heights compared to blackplate and lacquered ECCS.
4. The percentage of successfully drawn cups is greatest for film laminated materials relative to the other coated states, when forming a 0.26mm DR620 substrate over a 1mm die radius. This is attributed to over-stretching of the blank during forming in the uncoated variables.
5. The drawing operation is more likely to be unsuccessful at higher blank holder loads. This occurs in all coated states.
6. Cup height increases with increasing substrate thickness. A thicker blank has more material to be drawn and for a given set of tooling exhibits a greater tendency for lower wall thinning.
7. Cup height increases with increasing substrate thickness. However, this relationship is not linear and is approaching a limiting value at nominal 620MPa ultimate tensile strengths, for a draw ratio of 1.87:1.

Effect of polymer coating

8. Cups can be successfully drawn from all polymer coatings tested, with no adverse affect on coating integrity. This is true for all conditions investigated.
9. There is small difference in drawn cup dimensions between similar thickness polypropylene and polyethylene terephthalate coatings at very high blank holder forces.

10. Very little difference in drawn cup height dimensions was shown between 15 and 30μm polyethylene terephthalate coatings.
11. No difference in forming characteristics was evident between white and clear polymer coatings. This is true for both polypropylene and polyethylene terephthalate film laminates.
12. X-ray photoelectron spectroscopy and AFM analysis showed no TiO_2 pigmentation particles in the top few atomic layers of white polyethylene terephthalate laminated materials.
13. No change in cup height was found between polypropylene coated materials applied at different laminating temperatures.

Effect of tooling

14. Increasing blank holder force increases cup height. This is true for all materials investigated. This is attributed to a larger retardation force over the die radius at larger blank holder loads.
15. Higher blank holder forces produce cups with a greater degree of cup wall thinning.
16. Increasing blank holder force decreases the difference between average minimum and average maximum cup heights. Therefore, less material is lost through trimming to minimum cup heights.
17. Insufficient blank holder forces cause severe cup wall wrinkling and can result in irreparable tooling damage.
18. Too high a blank holder force causes tear-off of the sample in the region of the blank first in contact with the punch during the drawing operation.
19. The range of blank holder force, which can successfully be employed to produce a cup, has been termed the operating window. This window varies with substrate specification, coating type and die radius.
20. Increasing die radius results in a vertical shift in operating window. This is attributed to different energy loses over the die radius.
21. Film laminate materials can be manufactured over die radii of less than five times the total laminate thickness.
22. For 20μm polyethylene terephthalate materials a 1.75mm increase in cup height is noted between 2.0 and 1.0mm die radius.

23. A linear relationship exists between cut edge dimensions and cup height. This is true for all die radii and punch diameters investigated.
24. Extrapolation of cup height data for a range of cut edge sizes back to zero height does not show an intercept at the punch diameter dimension. Two reasons for this anomaly exist; material is gained over the punch nose in the first few millimetres of cup height and a greater degree of cup wall stretching is measurable at larger cut edge sizes.
25. A 0.18mm substrate with nominal ultimate tensile strength of 580MPa laminated with 20μm clear and 25 μm white polyethylene terephthalate shows a limiting draw ratio of approximately 2.0.
26. Increasing punch diameter decreases cup height for a given cut edge size.
27. Smaller punch diameters exhibit less cup wall thinning than large punch diameters.
28. Assuming uniform wall thickness and no substrate planar anisotropy (and hence earing), cup height can be derived as:

$$h = \frac{P_{dia}}{4}\left[D_{rat}{}^2 - 1\right]$$

29. Taking into account planar anisotropy and punch nose radius, this equation can be refined to give a 95% accuracy for all cut edge and punch diameters considered for a 0.18mm DR580 substrates to:

$$h = \frac{P_{dia}}{4}\left[D_{rat}{}^2 - 1\right] - \frac{P_{dia}}{8n}\left[D_{rat}{}^2 - 1\right] - \frac{c}{n} + \mathrm{P}_{\mathrm{r\,ad}}\left[2 - \frac{\pi}{2}\right]$$

Effect of lubrication

30. Lubrication levels per disc influence the drawn height of a cup. Higher lubrication weights produce a shorter cup. This reaches a limiting value at weights above 0.5mg per disc.
31. At very low lubrication levels discs are more likely to fail. Successful cups show greater variation in cup height than those produced at higher lubrication weights.

8.2 Biaxial stressing of film laminated materials

32. Newly laminated materials craze under the application of a biaxial stress field within one week of lamination.
33. Stoving of samples above the polymer coatings glass transition temperature eliminates craze formation for a period of time.
34. The maximum time period after stoving, which crazing is eliminated is three days at a stoving temperature of 90°C.
35. The likely mechanism for craze formation is one of environmental stress crazing. Although an interaction of ageing and crazing has been noted.
36. Very small dome simulation depths produce crazes in fully aged laminate material.
37. When crazing occurs it is a time dependant phenomenon, taking up to three hours after biaxial stressing to reach its final level.
38. The addition of Dioxylsebacate, Acetyltributylcitrate or Butylstearate oils to the surface of a laminate material prior to deformation dramatically increases the severity of crazing. A relationship between solubility parameters of the lubricants and polyethylene terephthalate has been noted.
39. Laminate material produced without the addition of rolling lubricants to the surface of the substrate prior to lamination does not craze at any time period after production.
40. The addition of a petrolatum wax to the surface of fresh laminate material delays the onset of severe crazing by up to one month.
41. Laminating preheat temperature regime was found to have no effect on craze formation or magnitude.

9 Recommendations for Further Work

The most obvious point to raise when considering future research in this area is redrawing of laminate materials. For ease of analysis and to allow a broader spectrum of investigation this research only considered single stage drawing of materials from a known cut edge dimension to a first cup. For all can making methods this cup requires considerable extra work to achieve the dimensions required for a final product. In the case of DWI can manufacture, the cup is ironed through a series of rings until the required height is reached. In DRD and aerosol manufacture the first stage cup is redrawn between three and five times before trimming, necking and doming take place. The subsequent redrawing or ironing of film laminated materials from the first cup state achieved in this document is an obvious area for expanded research. Careful consideration is required before exploration of this avenue, due to the complexities of redraw interactions and detection of problems produced in previous operations. However, this remains the single most important avenue to explore if laminate manufacturers and can makers are to full understand drawing characteristics of film laminated materials.

The research focussed entirely on double reduced (DR) substrates with a gauge range of 0.15 to 0.26mm and nominal ultimate tensile strengths of 520 to 620MPa. The gauge and strength spectrum investigated covers the vast majority of the DR substrate product range. However, very little assessment of single reduced (SR) substrates has been considered. Therefore, if the current commercial product range is to be further extended into film laminated SR substrates, for drawing operations, this area needs to be investigated.

Once a full understanding of forming properties of all substrate passivation, tempers and strengths is understood a database could be compiled. This could then be further enhanced by applying the findings of the current research regarding polymer coating additions and provide a useful tool for benchmarking of specific drawn applications.

The majority of polymeric coatings investigated were polyethylene terephthalate and polypropylene. However, variations on these exist such as, PET(g) a glycol

substituted PET coating and PPN, polypropylene with a nylon topcoat. Other additions are also possible; isophthalate to PET for example, to reduce crystallininty. These new coatings are not expected to vastly influence the results, but a certain amount of verification would be required to confirm this assumption.

To make the cup height predictive equation increasingly accurate, a better understanding of earing is necessary. Literature suggests earing is governed by chemistry and percentage reduction. This research has shown that draw ratio and blank holder fore also effect earing values. Therefore, large amounts of additional research are required to fully predict earing for a given material. A wider range of punch diameters or cut edge sizes would be required to fully analyse the assumption that the c constant is variable with either punch size or draw ratio.

Gridmarking of the dome simulations proved unsuccessful due to the low percentage of elongation to failure in double reduced materials. However, it would be useful to quantify strain to craze formation for a particular laminate material. Although, it has been shown production of dome simulations even at very low deformation depths exhibit crazing. It is unlikely that a dome profile could be found for the material investigated, which achieves the necessary strength and does not exhibit crazing. Nonetheless, it would be beneficial to can designers to have a critical strain value for crazing. To achieve this a measurement method would have to be developed.

The majority of work investigated in this document has been produced on the Ferrolite Line at Trostre works in South Wales. The methods of film laminating are different to Corus' sister plants in IJmuiden and Duffel. The IJmuiden method produces a product called Protact via a slightly different film application process. However, the Duffel methodology is entirely different. Polymeric coatings are directly extruded onto the steel substrate from a pelletised source. Therefore, in order to transfer findings of this research directly to the above materials a certain amount of verification work would be required. The main differences envisaged would be with the directly extruded material. Here the polymer is approximately 12μm in thickness compared to 20 to 40 μm for the investigated laminates. Provided the same polymer

structure is exhibited as in the materials investigated, the conclusion relating to polymer coating should remain valid. The other main area of anomaly between the polymer coated steel range is in the substrate employed. There are variations in both composition and physical properties of the substrate, when produced on different sites. In this instance large amounts of further research would be necessary to compare standard substrate properties and subsequent deformation behaviour. However, the exact results would be expected to change in the new product range, but the generic trends should still remain.

10 References

1. Encyclopaedia Britannica: 200th Anniversary Edition, 1970, Vol. 4, p787.
2. C. Archer, *Beverage Cans - The Future*, Soft Drinks Management International, Dec 1995, pp24-27.
3. J. Mack, *Lifting the Lid on Packaging*, Metal Bulletin Monthly, July 1996, pp36-39.
4. R. Opie, *Packaging Source Book*, MacDonald Orbis, London, 1989, pp86-146.
5. H. Houghton, *Packaging Closures*, Soft Drinks Management International, Apr 1994, pp25-30.
6. P. Jenkins, *Twenty by Fourteen: A History of the South Wales Tinplate Industry*, Gomer Press, Dyfed, 1995, p45.
7. P. Donaldson, *The Golden Beer Can*, Brewers Guardian, Nov 1985, pp20-21.
8. P. Marsal, *The Can and it's Uses. Part7a: Protect and Decoration of Cans by Lacquering*, The Canmaker, Aug 1989, pp39-42.
9. D.I.H. Johnstone, *Centenary Review: Beer Packaging in a Can*, Inst. Brewers, Nov-Dec 1986, Vol.92, pp529-536.
10. E. Morgan, *Tinplate and Modern Canmaking Technology*, Pergammon Press, Oxford, 1985, pp107-188.
11. J. Chevreux, *The Steel Beverage Can in Europe*, Soft Drinks Management International, Dec 1995, pp26-27.
12. The Metal Can. Open University Press, 1980.
13. C.G. Demmer, & N.S. Moss, A New Development in Water-Based Can Coatings, paper presented at OCCA-34 exhibition, 27-29 Apr 1982, pp249-256.
14. P. Newman, *Steel Versus Aluminium: The Battle Lines are Drawn*, The Canmaker, Oct 1992, pp32-38.
15. Clash of the Cans, Financial Times, 28 July 1993, p9.
16. P. Gander, *Easy Does It*, The Canmaker, Nov., 1996, pp28-32.
17. J. Nutting, *Canmakers Shaping up to Challenge*, The Canmaker, May 1996, p9.
18. B. Miyares, *Will You Find Profits as Well as Coke ?*, The Canmaker, March 1995, p14.
19. J. Ward, *Contoured Cans Could be Lighter*, The Canmaker, Nov. 1996, p14.
20. J. Nutting, *Reforming Zeal*, The Canmaker, Feb. 1996, pp32-37.
21. R. Goddard, *Tokyo Pack '94*, Canner, Oct. 1994, pp39-41.
22. J. Nutting, *Biting the Bullet*, The Canmaker, May 1997, pp54-58.
23. C. Moore & R.I. Marshall, *Steelmaking*, The Institute of Materials, 1991.
24. K.C. Barraclough, *Steelmaking 1850-1900*, The Institute of Materials, 1990.
25. A. Gnyp, *The lightweighting of Two Piece Beer Cans*, MBAA Technical Quarterly, Vol. 20, No.3, 1983, pp93-98.
26. B.L. Hitchins, & S.R. Postle, *Environmentally-Driven Advances in Can Coatings Technology*, Polymers Paint Colour Journal, July 22, 1992, Vol. 182, pp400-402.
27. F.L. Church, *New Can Coatings Avoid "Perils" of PVC*, Modern Metals, Feb 1992, 48 (1), pp44cc-44hh.
28. D.W. Fone, *Polymer Chemistry and Water-Borne Can Coatings*, Paper 21: presented at the Fourth International Tinplate Conference, London, 10-14 Oct 1988, pp227-236.
29. D.J. Barkel, *High Solids for Food Can Coatings*, Paper 20: presented at the Fourth International Tinplate Conference, London, 10-14 Oct 1988, pp219-226.
30. N. Kaguma, H. Iwashita & A. Tanaka, *Polyester Film Laminating Technology*, Paper 31, presented at the Sixth ITRI conference, London, 1996.
31. Packaging Week, May 25, 1988, p65.
32. N. Busby & B.J. Bastable, *"Ferrolite"- A Packaging Material for the Future*, Procs. Coatings in Packaging Conf., London, Nov 1998, Paper 3.

33. BST Brochure. Ferrolite: *A New Generation of Steel for the Packaging Industry*, Tin International, 1992, p65.
34. A.K. Biswas, *Principles of Blast Furnace Ironmaking*, Cootha Publishing House, 1981, p7.
35. R.D. Pehlke, et al. *BOF Steelmaking: Volume one*, Iron and Steel Society of the American Institute of Mining and Metallurgy and Petroleum Engineers Inc., 1974, p20.
36. J.D. Jevons, *The Metallurgy of Deep Drawing and Pressing*, Second Edition, Chapman & Hall Ltd, London, 1949, p183.
37. J. Nutting, *New Direct Extrusion Method Offers Improvements for Laminated Products*, The Canmaker, May 1999, p69.
38. S. Sakamotot, K.Kano & A. Tanaka, Changes of PET Film Structure in a Laminating Process, CAMP-ISIJ, Vol. 9, No. 376, 1996, pp 1246-9.
39. A. Tanaka, T. Hanafusu, H. Kojo & T. Inui, *The Adhesion of Biaxially Oriented Polyethelyene Terephthalate*, Testu to Hague (J. Iron & Steel Inst. Of Japan), 72, (8), 1986, pp301-308.
40. J Siewert, *Adrafol® – A Step Ahead*, Aerosol Spray Report, Vol. 30, No. 12, 1991, pp683-687.
41. Eds. *Canmaking System Saves 15%*, Packaging, Dec 1987, pp118-120.
42. S.C. Ball, D.T. Llewellyn & N. Jenks, *Effects of Coiling Temperature and Continuous Annealing Variables on Final Properties of Drawn and Wall Ironed Tinplate Steel*, Ironmaking and Steelmaking, Vol. 25, No. 1, 1998, pp55-62.
43. Y. Bergstrom, *'On the Strain Hardening Exponent n and the True Strain to Necking in Iron & Steel'*, Paper 1: Presented at the Eighth Biennial Congress the International Deep Drawing and Research Group, Gothenburg, Sweden, 23-27 Sept 1974, p4-26.
44. R.H. Wagoner & J-L. Chenot, *Fundamentals of Metal Forming*, John Wiley & Sons Inc., New York, 1997, p11.
45. P.C. Painter & M.M. Coleman, *Fundamentals of Polymer Science*, Technomic Publishing, Pennsylvania, 1994, p36.
46. E.A. Turi (ed), *Thermal Characterisation of Polymeric Materials*, Academic Press, New York, 1981, p242.
47. M. Hojo & H. Muroaka, *New PET Film for Laminated Cans*, Paper 19 presented at BEV-PAK Americas 1997, Florida, Apr 14-15, 1997, pp19.1-19.22.
48. H. Schrafft & J. Nutting, *The Big Squeeze*, The Canmaker, Oct 1995, pp41-42.
49. A.W. Birley, B. Haworth & J. Batchelor, *Physics of Plastics: Processing, Properties and Materials Engineering*, Hanser, New York, 1992.
50. T.S. Chow, *Stress Strain Behaviour of Physically Ageing Polymers*, Polymer, 1993, Vol. 34, No. 3, pp541-545.
51. E.J. Moskala, Environmental *Stress Cracking of PET Beverage Containers*, paper No. 8, presented at BEV-PAK Americas '96, Fort Lauderdale, Florida, USA. April 15-16, 1996.
52. L.C.E. Struik, *Physical Aging in Plastics and Other Glassy Materials*, Polymer Engineering & Science, March, 1977, Vol. 17, No. 3, pp165-173.
53. A.S. Kuz'minskii (ed), *The Aging and stabilisation of Polymers*, Elsevier Publishing, London, 1971.
54. J.C. Arnold, *The Influence of Physical Aging on the Creep Rupture Behaviour of Polystyrene*, J. of Polymer Science: Part B: Polymer Physics, Vol. 31, 1993, pp1451-1458.
55. J.C. Arnold, *The Effects of Physical Aging on the Brittle Fracture Behaviour of Polymers*, Polymer Engineering and Science, Vol. 35, No. 2, 1995, pp165-169.
56. P.M. Gezovich, & P.H. Geil, *Deformation and Ageing of Quenched Polypropylene*, Polymer Engineering and Science, Vol. 8, No. 3, 1968, pp210-215.
57. G.E. Sweet & J.P. Bell, *Aspects on the Yield Behaviour in Poly(ethylene terephthalate) films*, Polymer Engineering and Science, Vol. 19, No. 1, 1979, pp18-22.

58. P.H. Pfromm & W.J. Koros, *Accelerated Physical Ageing of Thin Glassy Polymer Films: Evidence from Gas Transport Measurements*, Polymer, Vol. 36, No. 12, pp2379-2387.
59. R.P. Kambar, *Stress-Strain Behaviour of the Craze*, Polymer Engineering and Science, Vol. 8, No. 4, 1968, pp281-289.
60. R.J. Crawford, *Plastics Engineering*, second edition, Pergammon Press, Oxford, 1992, p128.
61. E.J. Kramer, *Craze Fibril Formation and Breakdown*, Polymer Engineering and Science, Vol. 24, No. 10, 1984, pp761-769.
62. T. Yamamoto & H. Furukawa, *Relationship Between Molecular Strength and Deformation – Fracture Mechanism of Amorphous Polymers:2. Crazing Stress*, Polymer, Vol. 36, No. 12, 1995, pp2393-2396.
63. T. Juska & I.R. Harrison, *A Criterion for Craze Formation*, Polymer Engineering and Science, Vol. 22, No. 12, 1982, pp766-776.
64. I. Narisawaw & M. Ishikawa, *Crazing in Semicrystalline Thermoplastics*, Advances in Polymer Science 91/2: Crazing in Polymers Vol. 2, Springer-Verlag, London, 1990, pp353-393.
65. E.J. Kramer & L.L. Berger, *Fundamental Processes of Craze Growth and Fracture, Advances in Polymer Science 91/2: Crazing in Polymers Vol. 2*, Springer-Verlag, London, 1990, pp5-70.
66. M. Sugimoto, M. Ishikawa & K. Hatada, *Toughness of Polypropylene*, Polymer, Vol. 36, No. 19, 1995, pp3675-3682.
67. M. Ishikawa, K. Ushui & Y. Kondo, *Effect of Tie Molecules on the Craze Strength of Polypropylene*, Polymer, Vol. 37, No. 24, 1996, pp5375-5379.
68. P.L. Soni & P.H. Geil, *Environmental Stress Cracking of PET: Temperature Effect*, J. Applied Polymer Science, Vol. 23, 1979, pp1167-1179.
69. A. Jobanin E.A. Lofgren, *Environmental Ageing & Stress Cracking of PET*, Polymer Engineering & Science, Vol. 32, No. 2, 1992, p146
70. R. Fraser, *Environmental Stress Cracking of Plastics*, Plastics & Polymers, June 1975, pp12-13.
71. R. Qian, X. Lu & N. Brown, *The Effect of Concentration of an Environmental Stress Cracking Agent on Slow Crack Growth in Polyethylene*, Polymer, Vol. 34, No. 22, 1993, pp4727-4731.
72. M. Kawagoe & S. Nunomoto, *Crazing and Fatigue of Poly(methyl methacrylate) Due to Case II Diffusion of Methanol*, Polymer, Vol. 32, No. 17, 1991, pp3130-3135.
73. C.H.M. Jacques & M.G. Wyzgoski, *Prediction of Environmental Stress Cracking of Polycarbonate from Solubility Considerations*, J. Applied Polymer Science, Vol. 23, 1979, pp1153-1166.
74. P.I. Vincent & S. Raha, *Influence of Hydrogen Bonding on Crazing and Cracking of Amorphous Thermoplastics*, Polymer, Vol. 13, 1972, pp283-287.
75. Y.W. Mai, *Environmental Stress Cracking of Glassy Polymers and Solubility Parameters*, J. Materials Science, Vol. 21, 1986, pp904-916.
76. W. Panknin, *Principles of Drawing and Wall Ironing for the Manufacture of Two-Piece Cans*, ASM Materials Conference Paper No, 74, p16, 1977.
77. S.Y. Chung & H.W. Swift, *Cup Drawing from a Flat Blank: Part 1. Experimental Investigation*, Proc. Inst. Mech. Eng., 165, pp199-211, 1952.
78. M. Gotoh, M. Kotoh & M. Yamashita, *Studies of Stretch Drawing Process of Sheet Metal*, J. Materials Processing Technology, 63, pp123-128, 1997.
79. P.B. Mellor, *Sheet-Metal Forming*, International Materials Reviews, 1, pp1-19, 1981.
80. S. Qin, H.M. Shang, C.J. Tay & J.X. Mo, *On the Extent of Stretching During Deep Drawing*, J. Materials Processing Technology, 59, pp386-390, 1996.
81. P. Bruton, Crazing of Ferrolite, British Steel Internal Report, Technical note TRL/TN/684/98/D, July 1998.

82. M. Ishikawa & A. Tanaka, *Investigation on Method of Measuring Adhesion Strength Between Laminated PET Film and Surface Treated Steel Sheet*, CAMP-ISIJ, Vol. 9, 1996, p463.
83. M. Gotoh, M. Ishikawa & A. Tanaka, *Effect of Annealing Temperature on Adhesion of Polyester Film to TFS*, CAMP-ISIJ, Vol. 9, Paper 375, 1996, p1245.
84. M.A.H. Van der Aa, *Wall Ironing of Polymer Coated Steel Metal*, Universiteitsdrukkerij, TU Eindhoven, 1999.
85. H.C.E. Van der Aa, *An Experimental and Numerical Study of the Wall Ironing Process of Polymer Coated Steel Metal*, MT reportnr 98.029, Eindhoven University of Technology, 1998.
86. I. Fujiyama, *Press Formability of Organic coated Steel Sheet with Excellent Lubrication for Automotive Body Panels*, CAMP-ISIJ, Vol. 11, 1998, p545.
87. H. Hayashi, *Press Formability of Steel/Plastic/Steel Composite Sheets*, International Deep Drawing Research Group, May 1987.
88. X. Cheng & Zhang, *Research on the Forming Characteristics of Laminated Damping Sheets for Deep Drawing*, 16th Biennial Congress International Deep Drawing Research Group: Sheet Metals in Forming Processes, June 1990, pp215-221.
89. M. Shinozaki, & Y. Matsumoto, *Formability of Steel-Plastic-Steel Laminate*, 14th IDDRG Biennial Congress, Koln, April 1986, pp159-167.
90. N.D. Jenks, Technical Note No. TRL/TN/706/98/D, British Steel Tinplate, 18 December, 1998.
91. P.I. Vincent & S. Raha, *Influence of Hydrogen Bonding on Crazing and Cracking of Amorphous Thermoplastics*, Polymer, Vol. 13, 1972, pp283-287.
92. R. Organ, *Can Shaping – Metal at Its Forming Limits*, Paper 9, Session 2.7: Present and Future Challenges in Packaging materials, Materials Congress 2000, Cirencester, April 2000.
93. R.H. Wagoner (ed), *Forming Limit Diagrams: Concepts, Methods and Applications*. The Minerals, Metals and Materials Society, 1989.
94. G. LeRoy & J.D. Embury, *The Utilization of Failure Maps to Compare the Fracture Modes Occurring in Aluminium Alloys*, Paper 13, Procs of Symposium on Formability: Analysis, Modelling & Experimentation, Illinois, Oct 1977, pp183-208.
95. C.Sudo, Y. Hirose & Y. Hayashi, *Super Formative Steel Sheet with Excellent Stretchability and Low Effective Yield Strength*, Paper presented at 16th Biennial Congress IDDRG: Sheet Metals in Forming Processes, Borlänge, June 1990, pp391-403.
96. H.M. Shang, S. Quin and J. Mo: *J. Materials Processing Technology*, 1996, 62, pp454-457.
97. K. Manobe, H. Hamomo & H. Nishimura, *A New Variable Blank Holder Force Method in deep drawing of Sheet Metals*, CAMP-ISIJ Vol. 29, No 330, 1987, pp740-747.
98. Corus Packaging Plus quality document, QA2000, located in room EN1, Welsh Technology Centre.
99. J. Barndrup & E.H. Immergut, *Polymer Handbook*, Second edition, John Wiley & Sons, 1975.
100. S. Laurence, British Steel Internal Report, Technical note TRL/TN/665/97/D, Oct 1997.
101. R.C. Hudd, & K. Lyons. *Effect of Cup Ear Geometry and Thickening in Cylindrical Flat-bottomed Steel Cups*, Metals Technology, Sept 1975, pp428-432.
102. T.S. Chow, *Stress Strain Behaviour of Physically Ageing Polymers*, Polymer, 1993,Vol. 34, No. 3, pp541-545.
103. A. Lee & J. McGary, J. Materials Science, Vol. 26, 1988, p1313.
104. C. Bauwens-Crowet & J.C. Bauwens, *Annealing of Polycarbonate Below the Glass Transition – Quantitative Interpretation of the Effects on Yield Stress and Differential Scanning Calorimetry Measurements*, Polymer, Vol. 23, 1982, p1599

105. L.J. Broutman & S.M. Krishnakumar, Polymer Engineering & Science, Vol. 16, 1976, p74.
106. J.C. Arnold, *The Effects of Diffusion on ESC initiation in PMMA*, J. Materials Science, Vol. 33, 1998, p5193.
107. K. Tonyali, C.E. Rogers & H.R. Brown, *Stress Cracking of High Density Polyethylene in Detergents*, Polymer Engineering & Science, 1987, p2782.
108. A.F.M. Barton, *CRC Handbook of Solubility Parameters and Other Cohesion Parameters*, CRC Press, Boca Raton, 1983.

Composition	Weight %
Carbon	0.03 – 0.09
Manganese	0.20 – 0.30
Nickel	< 0.08
Copper	< 0.06
Chromium	< 0.06
Aluminium	0.03-0.07
Phosphorus	< 0.02
Sulphur	< 0.02
Silicon	< 0.02
Nitrogen	< 0.006

Table 2.1. Acceptable compositional range for a low-carbon mild steel

No.	Substrate			Coating top			Coating bottom			Total laminate thickness (mm)	Laminating conditions
	Thickness (mm)	Passivation	UTS (MPa)	Type	Thickness (μm)	Colour	Type	Thickness (μm)	Colour		
1	0.17	ECCS	620	PET	15	Clear	PET	15	Clear	0.20	Standard
2	0.195	Blackplate	620	n/a	n/a	n/a	n/a	n/a	n/a	0.195	n/a
3	0.195	ECCS	620	Lacquer	5	n/a	Lacquer	5	n/a	0.205	n/a
4	0.195	ECCS	620	PET	25	White	PET	20	Clear	0.24	Standard
5	0.26	Blackplate	620	n/a	n/a	n/a	n/a	n/a	n/a	0.26	n/a
6	0.26	ECCS	620	Lacquer	5	n/a	Lacquer	5	n/a	0.27	n/a
7	0.26	ECCS	620	PET	25	White	PET	20	Clear	0.305	Standard
8	0.19	ECCS	520	PET	25	White	PET	20	Clear	0.235	Standard
9	0.19	ECCS	580	PET	25	White	PET	20	Clear	0.235	Standard
10	0.15	ECCS	620	PET	25	White	PET	20	Clear	0.195	Standard
11	0.24	ECCS	620	PET	25	White	PET	20	Clear	0.285	Standard
12	0.18	ECCS	580	PET	25	White	PET	20	Clear	0.225	Standard
13	0.18	ECCS	580	PP	20	Clear	PET	20	Clear	0.22	Standard
14	0.18	ECCS	580	PP	40	White	PP	40	Clear	0.26	Standard
15	0.17	ECCS	580	PET	30	Clear	PET	30	Clear	0.23	Standard
16	0.18	ECCS	580	PP	40	White	PP	40	Clear	0.26	High

Table 4.1. Materials for investigation: drawing of film laminated materials.

	Internal die diameter (D_{dia}) (mm)	Die radius (D_{rad}) (mm)
A	67.246	1.68
B	67.488	1.0
C	67.488	1.36
D	67.488	2.0
E	89.45	1.27
F	89.45	2.54
G	91.04	3.175

Table 4.2. Available die tooling for use on the Roell & Korthaus cupping press

	External punch diameter (P_{dia}) (mm)	Punch nose radius (P_{rad}) (mm)
I	66.79	1.20
II	88.90	1.524
III	88.90	2.032
IV	90.42	3.175
V	66.79	Dome tooling

Table 4.3. Available punch tooling for use on the Roell & Korthaus cupping press

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Transverse	648	652	1.55
	Longitudinal	568	570	1.72
2	Transverse	647	667	1.57
	Longitudinal	568	571	1.92
3	Transverse	661	669	1.51
	Longitudinal	575	584	2.07
4	Transverse	649	666	1.54
	Longitudinal	580	583	1.4
Average	*Transverse*	*652.1*	*661.0*	*1.66*
	Longitudinal	*571.3*	*577.0*	

Table 4.4. Centre-line tensile measurements taken transverse and longitudinal to the rolling direction for 0.17mm DR620 material.

Disc number	Three drops per disc (mg)	Six drops per disc (mg)	Flooded (mg)
1	0.40	0.78	2.01
2	0.42	0.96	1.76
3	0.43	0.84	2.23
4	0.46	0.83	2.06
5	0.39	0.94	2.34
6	0.48	0.96	1.89
7	0.47	0.76	2.10
8	0.42	0.82	2.24
9	0.45	0.86	2.37
10	0.48	0.84	1.99
11	0.42	0.79	2.03
12	0.43	0.83	2.16
13	0.49	0.95	2.30
14	0.47	0.86	1.98
15	0.45	0.89	2.09
Average	*0.444*	*0.861*	*2.103*

Table 4.5. Lubrication weights for three sets of discs used to establish consistency in application method.

No.	Substrate			Coating top			Coating bottom			Total laminate thickness (mm)	Laminating conditions
	Thickness (mm)	Passivation	UTS (MPa)	Type	Thickness (μm)	Colour	Type	Thickness (μm)	Colour		
1	0.17	ECCS	620	PET	15	Clear	PET	15	Clear	0.20	Standard
2	0.17	ECCS	620	PET	20	Clear	PET	25	white	0.215	Standard
3	0.17	Blackplate	620	PET	20	Clear	PET	25	White	0.215	Standard

Table 4.6. Materials for investigation: biaxial stressing of film laminated materials.

Dome height (mm)	Overall craze dimensions (mm)	Crazed area ($x10^{-3}$ m^2)
0.5	N/A	-
1.0	N/A	-
1.5	Not measurable	-
2.0	6 x 6	0.12
2.5	11 x 11	0.38
3.0	13 x13	0.53
3.5	15 x15	0.71
4.0	18 x 17	0.96
5.0	25 x 23	1.81
7.0	35 x32	3.52
9.5	Failure	Failure

Table 4.7. Dome height deformation and crazed area dimension for using the standard experimental procedure.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	587	589	1.53
	Centre	583	586	1.72
2	Edge	590	592	1.43
	Centre	577	581	1.92
3	Edge	592	594	1.40
	Centre	579	583	1.99
4	Edge	593	595	1.54
	Centre	582	586	1.92
	Average	*585.4*	*588.3*	*1.68*

Table 5.1. Tensile properties at random locations within the tested material for the 0.195mm DR620 substrate.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	590	593	1.55
	Centre	589	593	1.72
2	Edge	592	594	1.52
	Centre	588	593	1.74
3	Edge	589	592	1.63
	Centre	588	592	1.71
4	Edge	589	593	1.62
	Centre	587	591	1.68
	Average	*589.0*	*592.6*	*1.65*

Table 5.2. Tensile properties at random locations within the tested material for the 0.26mm DR620 substrate.

Nominal Strength (MPa)	Properties		
	0.2% Proof stress	UTS	Elongation
520	520 ± 50 MPa	530 ± 50 MPa	0 - 2.5%
580	580 ± 50 MPa	590 ± 50 MPa	0 - 2.5%
620	620 ± 50 MPa	630 ± 50 MPa	0 - 2.5%

Table 5.3. Acceptable mechanical properties for a range of double reduced packaging grade steels.

Material	Number of discs	Number of successful cups	Failure rate
Blackplate	25	12	52%
Lacquer	25	24	4%
Ferrolite	40	39	2.5%

Table 5.4. Failure rates for 0.195mm substrate.

Material	Number of discs	Number of successful cups	Failure rate
Blackplate	25	10	60%
Lacquer	25	20	20%
Ferrolite	40	38	3%

Table 5.5. Failure rates for 0.26mm substrate.

Substrate	Coating	Minimum	Maximum	Average
0.195mm	Blackplate	0.294	0.444	*0.362*
	Ferrolite	0.1	0.138	*0.117*
	Lacquer	0.095	0.113	*0.103*
0.26mm	Blackplate	0.242	0.408	*0.291*
	Ferrolite	0.132	0.173	*0.164*
	Lacquer	0.105	0.132	*0.121*

Table 5.6. Measured co-efficient of friction for a selection of materials measured using an Altek machine on a 2kg setting.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	595	597	0.57
	Centre	579	580	0.82
2	Edge	594	597	0.55
	Centre	577	581	0.73
3	Edge	598	600	0.65
	Centre	577	583	0.99
4	Edge	593	594	0.99
	Centre	580	581	1.02
	Average	*586.6*	*589.1*	*0.79*

Table 5.7. Tensile properties at random locations within the tested material for the 0.15mm DR620 substrate.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	593	596	-
	Centre	585	589	1.19
2	Edge	589	592	-
	Centre	585	588	1.54
3	Edge	595	597	0.89
	Centre	587	590	1.02
4	Edge	592	596	1.00
	Centre	586	589	1.63
	Average	*589.0*	*592.1*	*1.21*

Table 5.8. Tensile properties at random locations within the tested material for the 0.24mm DR620 substrate.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	505	512	1.63
	Centre	497	502	3.43
2	Edge	504	508	2.25
	Centre	493	497	4.16
3	Edge	503	507	-
	Centre	497	501	3.68
4	Edge	508	509	-
	Centre	499	505	2.16
	Average	*500.8*	*505.1*	*2.89*

Table 5.9. Tensile properties at random locations within the tested material for the 0.19mm DR520 substrate.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	556	560	0.96
	Centre	565	567	1.43
2	Edge	561	584	0.85
	Centre	565	568	2.02
3	Edge	556	559	0.78
	Centre	555	558	1.42
4	Edge	558	560	0.89
	Centre	559	562	1.25
	Average	*559.4*	*562.3*	*1.41*

Table 5.10. Tensile properties at random locations within the tested material for the 0.19mm DR580 substrate.

Element	White coating	Clear coating
Carbon	70.7	71.0
Oxygen	29.0	29.0
Fluorine	0.3	< 0.1
Titanium	< 0.1	< 0.1
Nitrogen	< 0.1	< 0.1
Silicon	< 0.1	< 0.1
Total	*100.0*	*100.0*

Table 5.11. Surface compositional analysis of white and clear coatings of a film laminated material using XPS.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	567	571	1.40
	Centre	587	588	1.68
2	Edge	574	576	1.28
	Centre	599	603	1.53
3	Edge	580	582	0.98
	Centre	597	600	1.03
4	Edge	589	593	1.52
	Centre	601	602	1.75
	Average	*586.7*	*589.4*	*1.396*

Table 5.12. Tensile properties at random locations within the tested material for the 0.17mm DR620 substrate.

Sheet	Location	0.2% Proof Stress (MPa)	UTS (MPa)	Elongation %
1	Edge	557	561	1.38
	Centre	562	564	1.76
2	Edge	547	550	1.60
	Centre	568	572	1.50
3	Edge	565	569	1.38
	Centre	578	581	1.41
4	Edge	550	554	1.40
	Centre	579	580	1.69
	Average	*563.3*	*566.4*	*1.515*

Table 5.13. Tensile properties at random locations within the tested material for the 0.17mm DR620 substrate used for the blackplate based film laminate material.

	PET	BSO	ATBC	DOS
V (mol. Volume)	208.7	371.9	366.6	447.5
δ_d (dispersive)	18.5	17.1	16.7	17.1
δ_p (polar)	3.4	1.3	2.7	1.3
δ_h (hydrogen)	8.2	4.3	8.7	5.6
δ_d (total)	20.5	17.7	19.0	18.0

Table 6.1. Solubility parameters for PET and lubricants used in ESC analysis.

Year	Key Development
1795	French government offer FFr. 12,000 for discovery of food preservation method
1809	Nicholas Appert develops sterilisation process
1810	Peter Durand granted patent for tinplated iron food container
1846	Henry Evans invents device to manufacture a can in a single operation
1880	Automated can making machine introduced to Britain
1891	William Painter develops the 'crown cork seal' airtight lid
1914-1918	Great war
	Food rationing decline in packaging developments
1935	First alcoholic beverage packed in Felinfoel, Llanelli
1930s	Drinks packaged directly into three-piece tinplate cans
	Flat top cans developed – easily stackable and transportable
	Variety of lacquers/linings were introduced to extend canned beer shelf life
1939-1945	Second World War
1940s	Label sizes reduced
	Food rationing
	Public beer consumption halted
1953	Rationing ended
1950s	Electrolytic tinning introduced to Britain
	Domestic refrigeration introduced, food life increased, chilled drinks available
	TV advertising to differentiate products – vast printing developments
	Carbonated soft drinks introduced and become popular with teenagers
1963	Easy open end ring-pull developed
1964	Drawn and wall ironed technology developed by Reynolds Metals Co.
	Necked in can produced to 2"9/16 diameter using a die flanger
1960s	Oleo-resins developed, good barrier properties, but not formable
	Epoxy phenolic lacquers, improved barrier properties, High VOC content
	Tin free steel developed in Japan to compete with tinplate
1970s	Draw redraw process developed
	Water based lacquers introduced
1980s	Two-piece beverage can market dominate by aluminium
	Necked in 2"6/16 can developed
	Stay on ring-pull use widespread
	Steel polymer laminates developed
	Metpolam licensed by British Steel from Metal Box
1992	Metpolam named Ferrolite
	Aluminium polymer steel 'Alulite' launched by CMB
	British Steel, Hoogovens and Rasselstein develop an all steel ultimate can
1993	Steel end named 'Ecotop' launched
1994	Coca-Cola advise all its suppliers that it is moving away from PVC based coatings
	Shaped Coca-Cola can tested in Germany and SE Asia
1997	British Steel Tinplate and Redicon launch RBS project, a Ferrolite cans

Figure 2.1. Chronology of key packaging influences and developments 1795-2000.

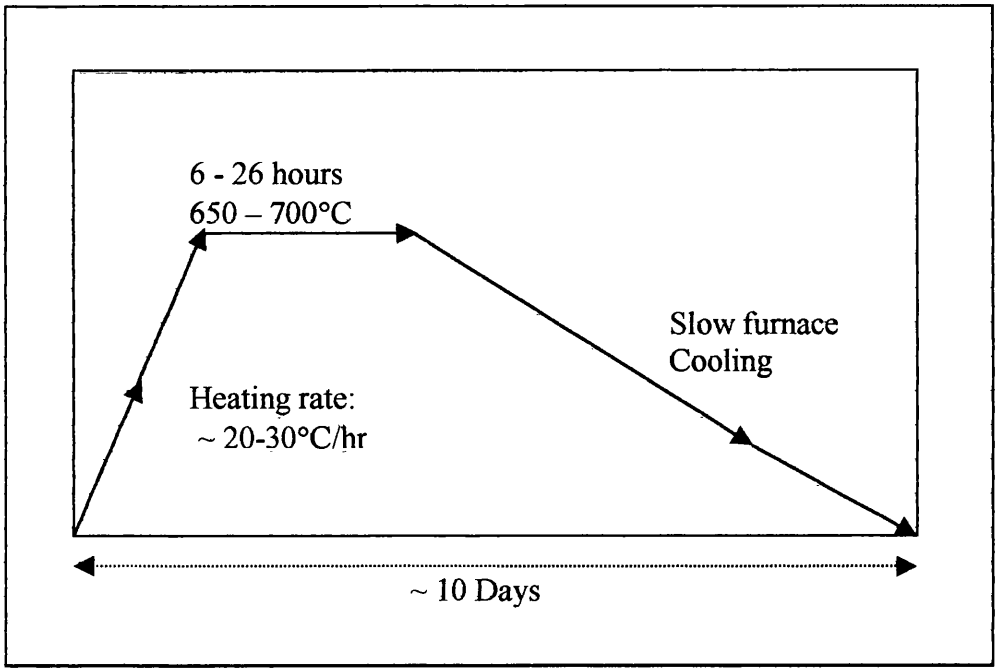

Figure 2.2. A typical batch annealing cycle used in production of packaging grade steel substrates.

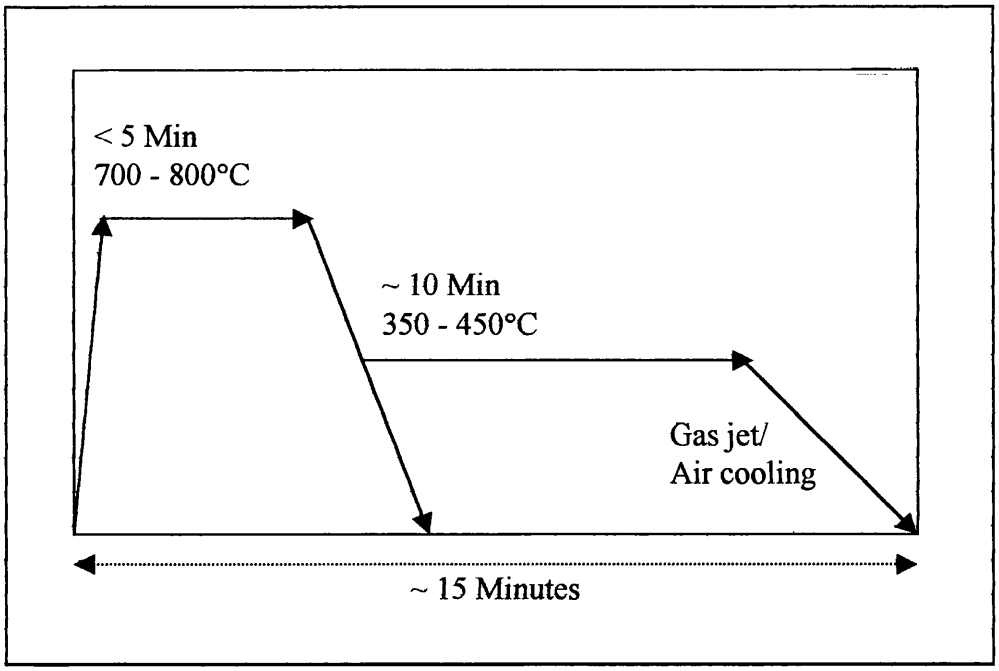

Figure 2.3. A typical continuous annealing cycle used in production of packaging grade steel substrates.

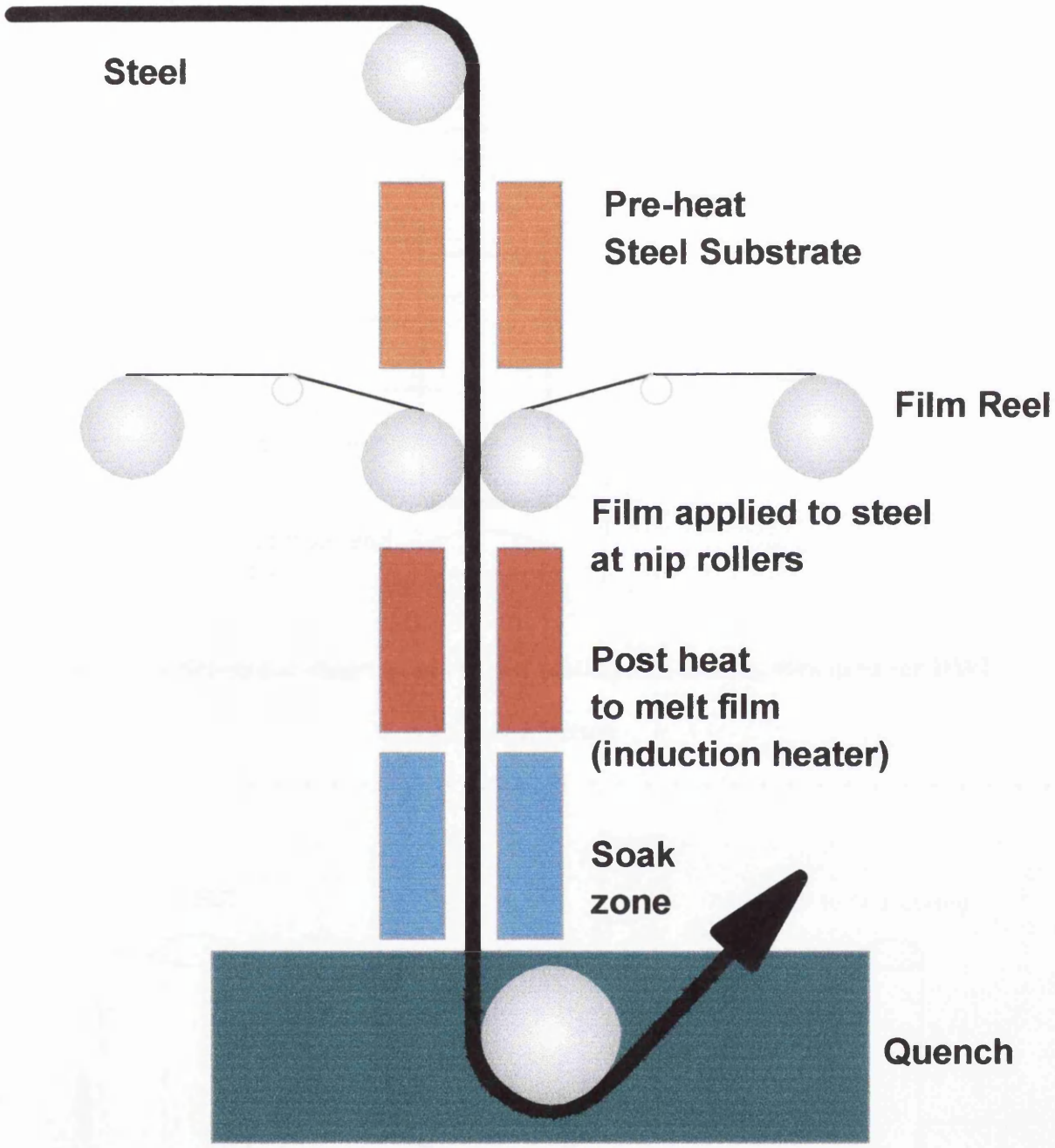

Figure 2.4. Schematic diagram of the Ferrolite film application process.

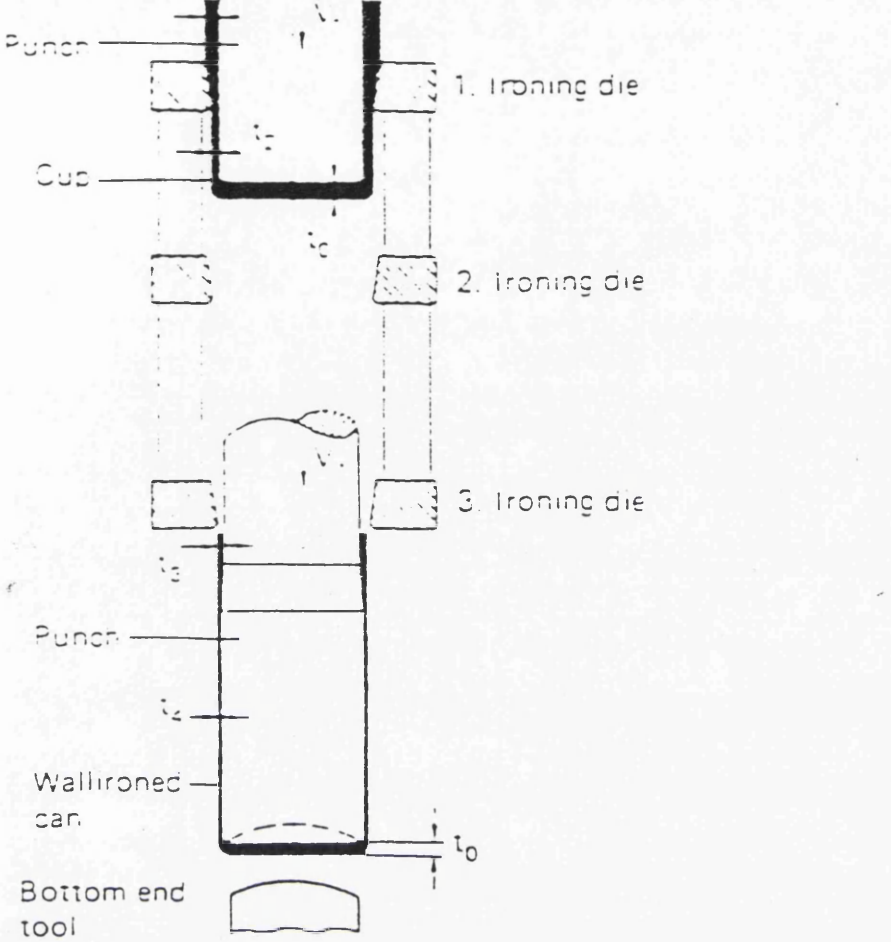

Figure 2.5. Schematic diagram of redraw tooling and ironing dies used for DWI can manufacture

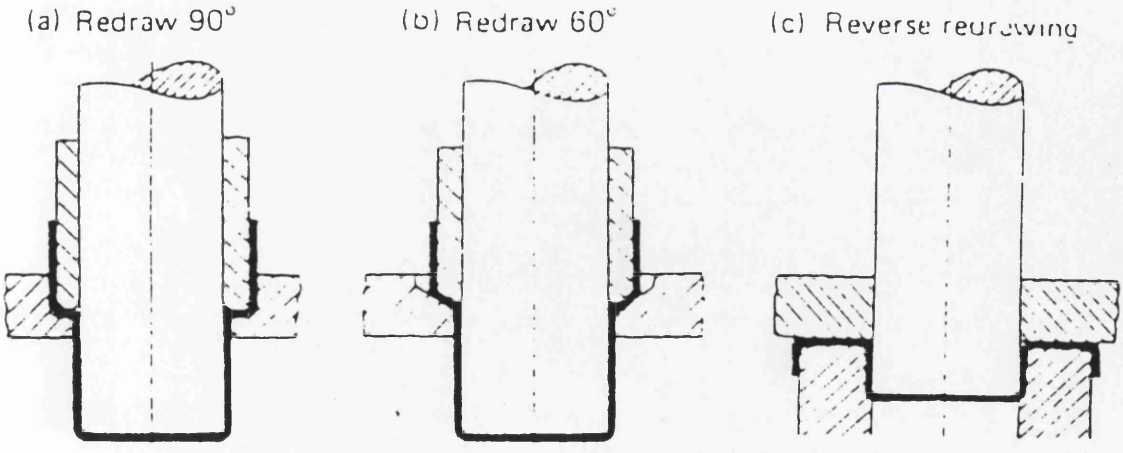

Figure 2.6. Schematic diagram of redraw tooling used for DRD can manufacture

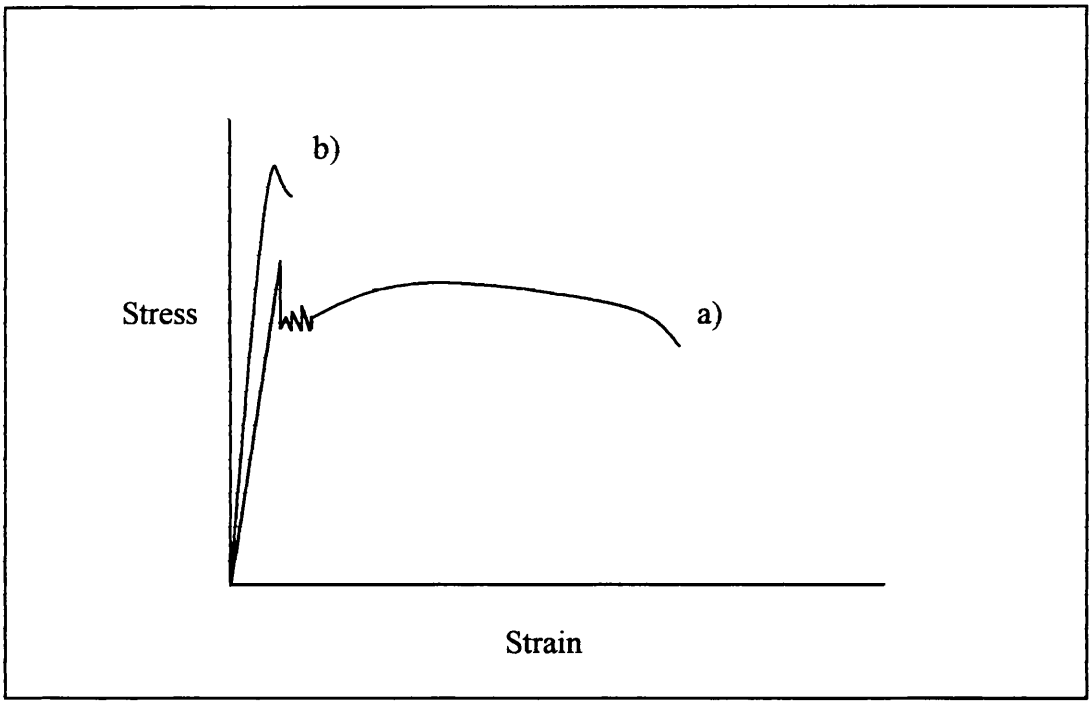

Figure 2.7. Representative tensile curves for packaging grade materials

a) Single reduced

b) Double reduced

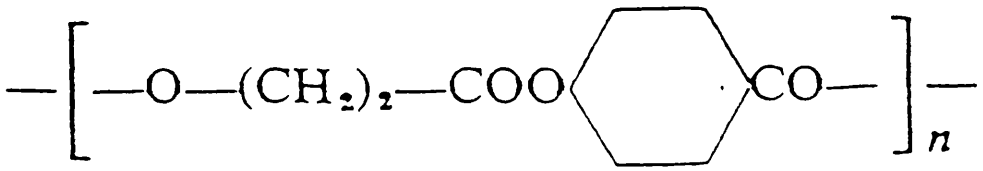

Figure 2.8. Polyethylene terephthalate repeat unit

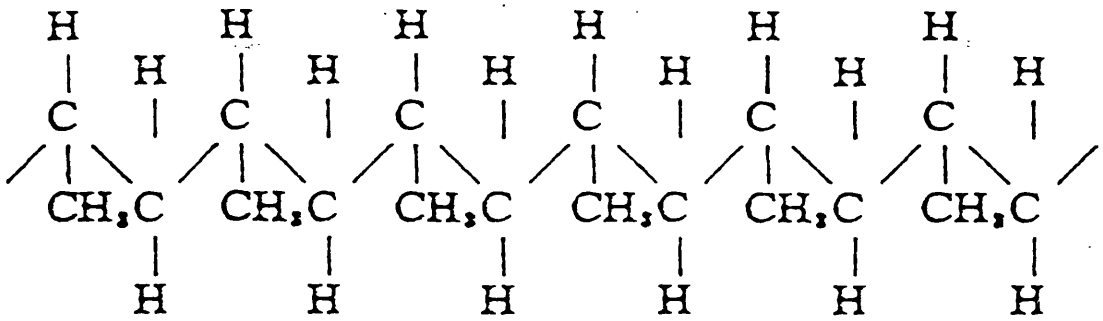

Figure 2.9. Polypropylene polymer chain

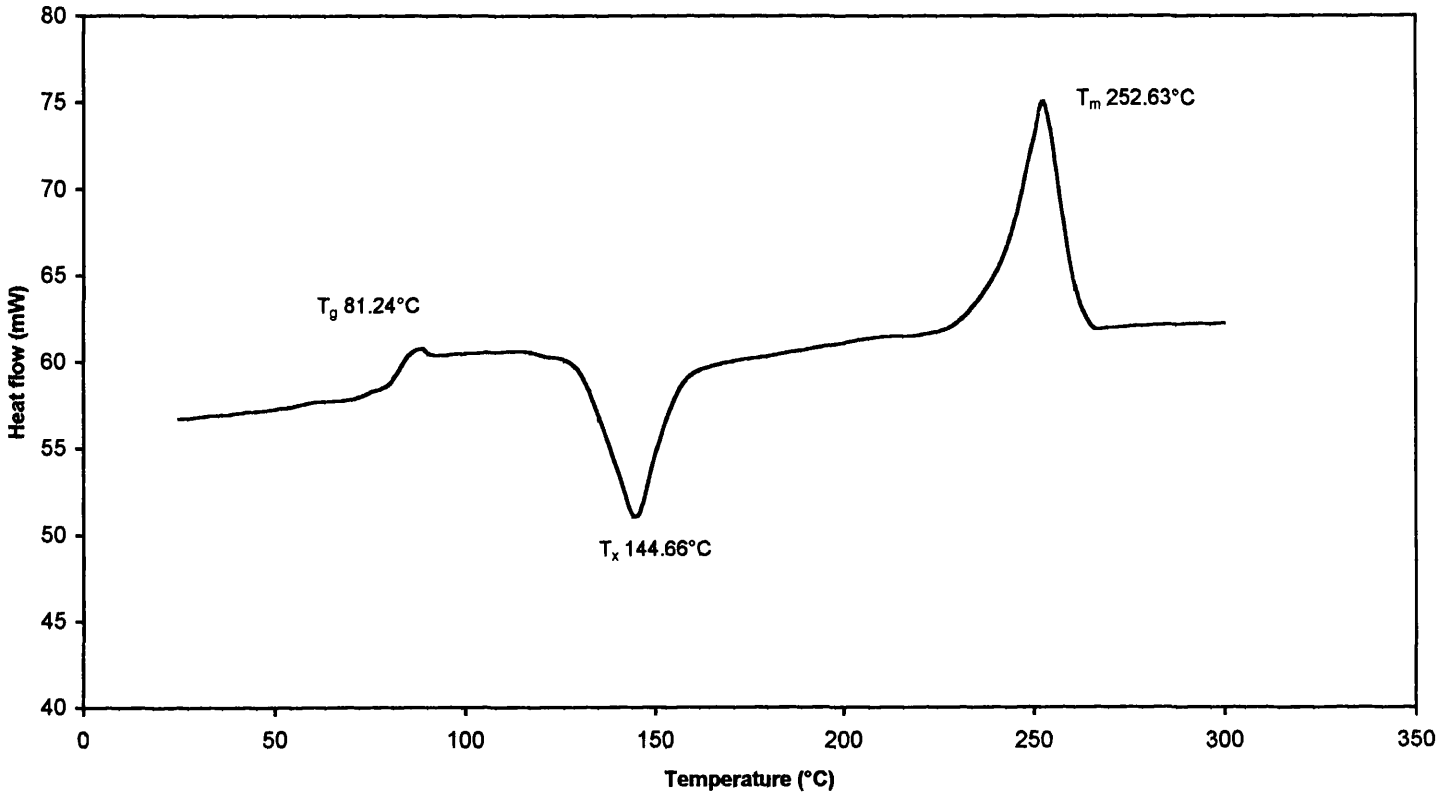

Figure 2.10. Differential scanning calorimetry trace showing changes in enthalpy occuring during heating of a Polyethylene Terephthalate film from ambient temperatures.

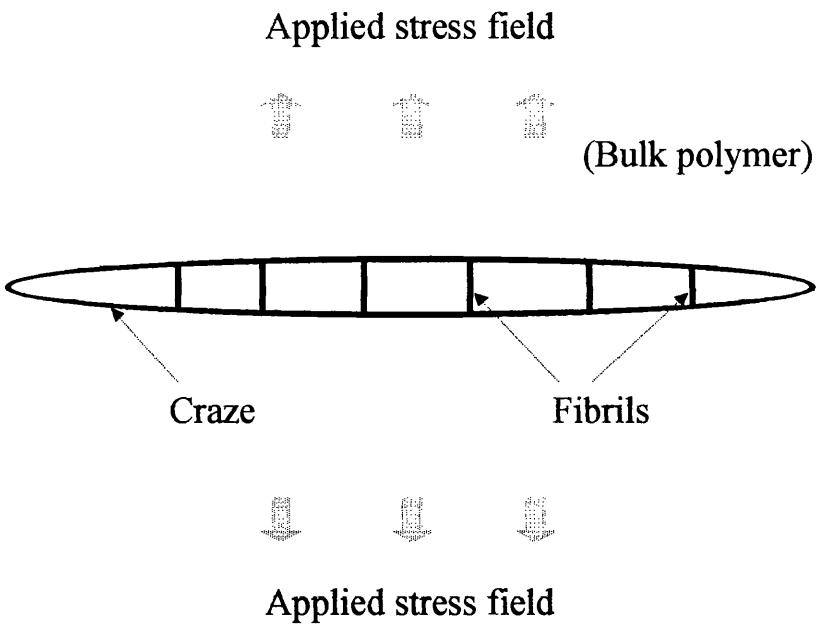

Figure 2.11. Schematic diagram of a craze and associated fibrils

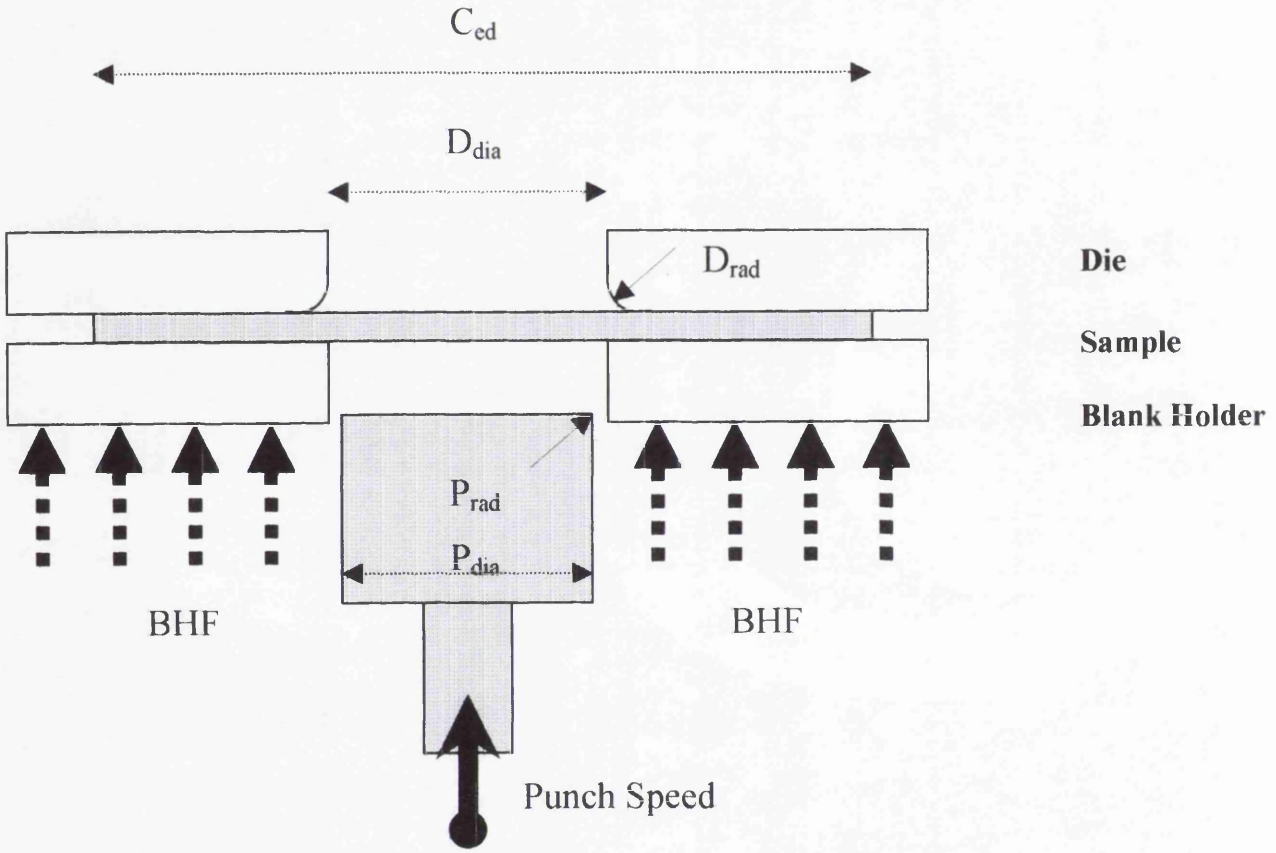

Figure 2.12. Schematic diagram showing critical parameters during formation of a cup from a flat disc

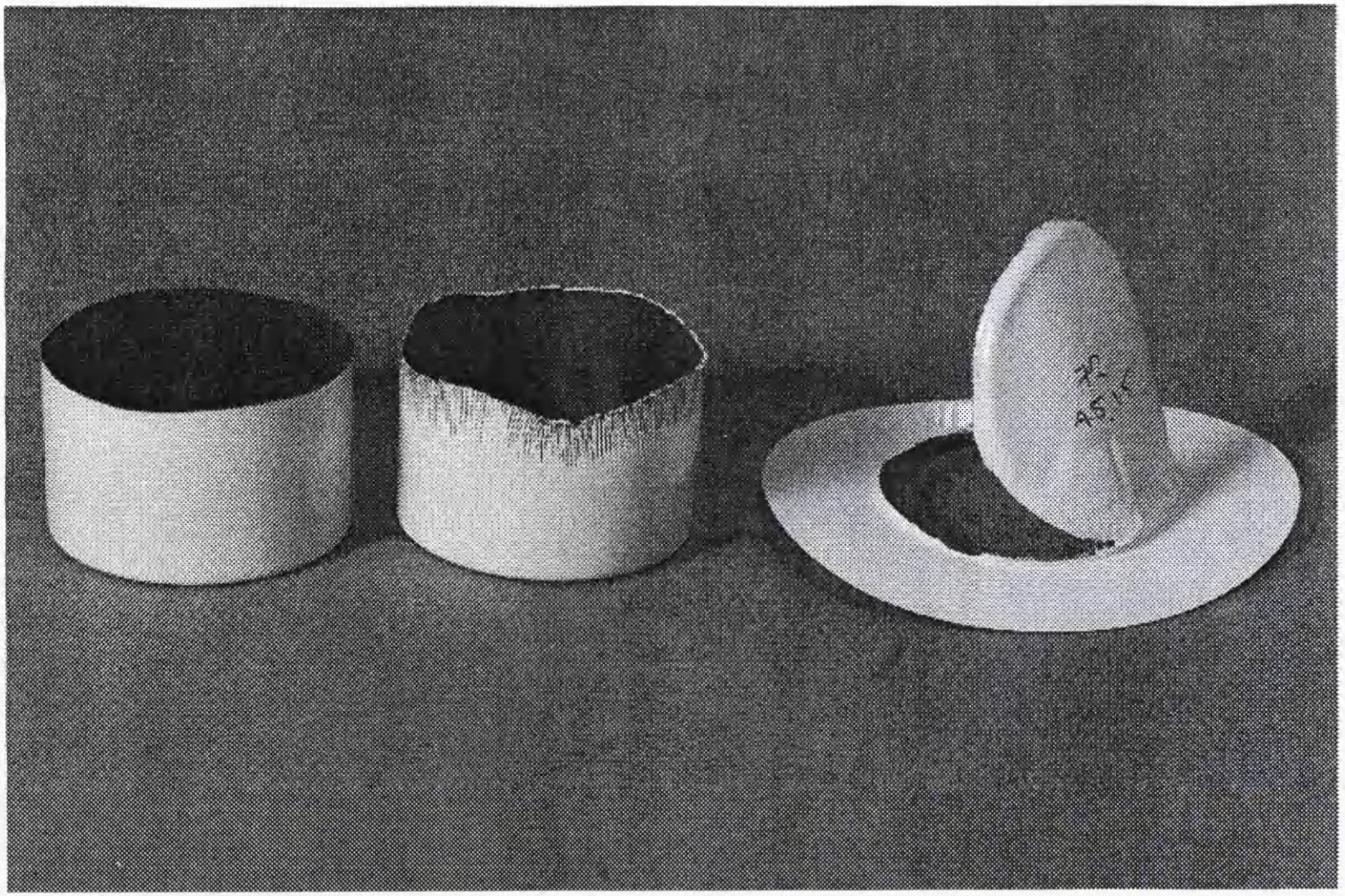

Figure 2.13. Cups produced over a range of blank holder forces

Left: cup made with acceptable blank holder force

Centre: excessive wrinkling caused by insufficient blank holder force

Right: tear-off caused by too high a blank holder force

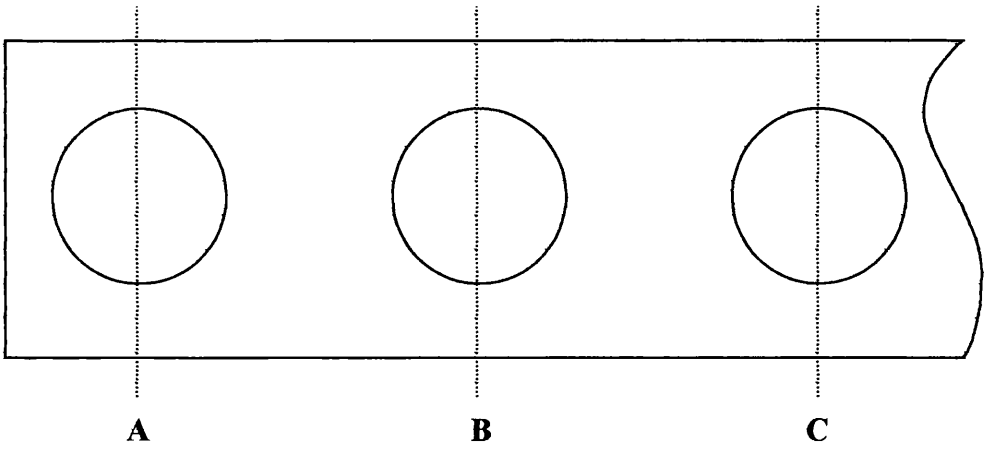

Figure 4.1. A schematic diagram indicating one half of tested sample positions across a coil width.

A – Centre of disc located 65mm from edge of coil

B – Centre of disc located midway between discs A and C

C – Centre of disc located 65mm from centre of coil

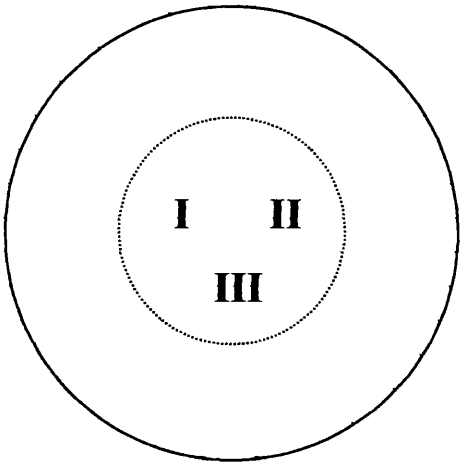

Figure 4.2. Disc sample showing nomenclature for cupping experiments.

I – Indicates position of disc within the coil

II – Indicates the cupping order

III – Indicates the required blank holder force (if different from standard)

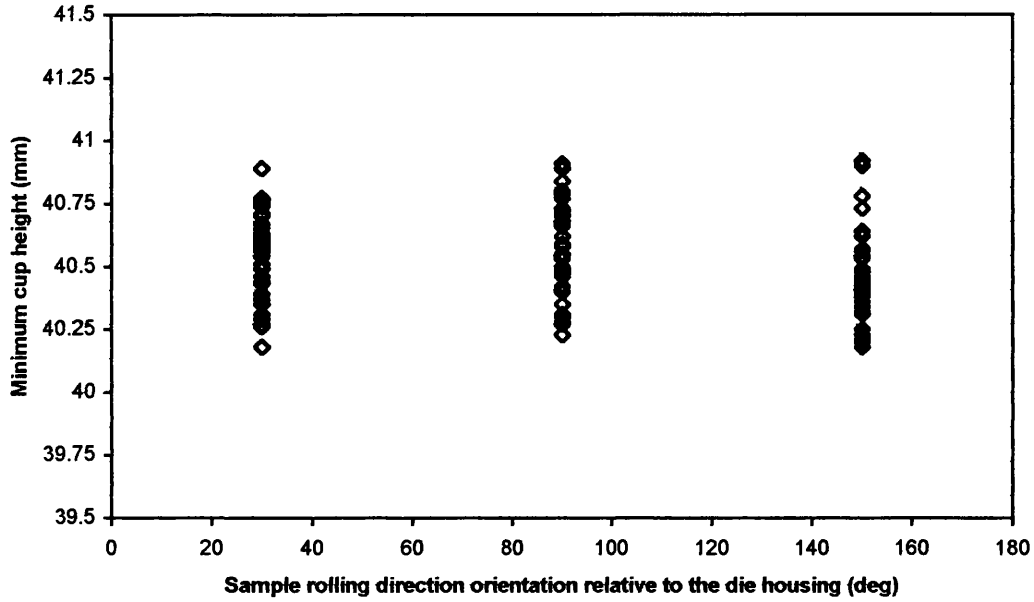

Figure 4.3. The influence of sample rolling direction in relation to the die housing on drawn minimum cup height.

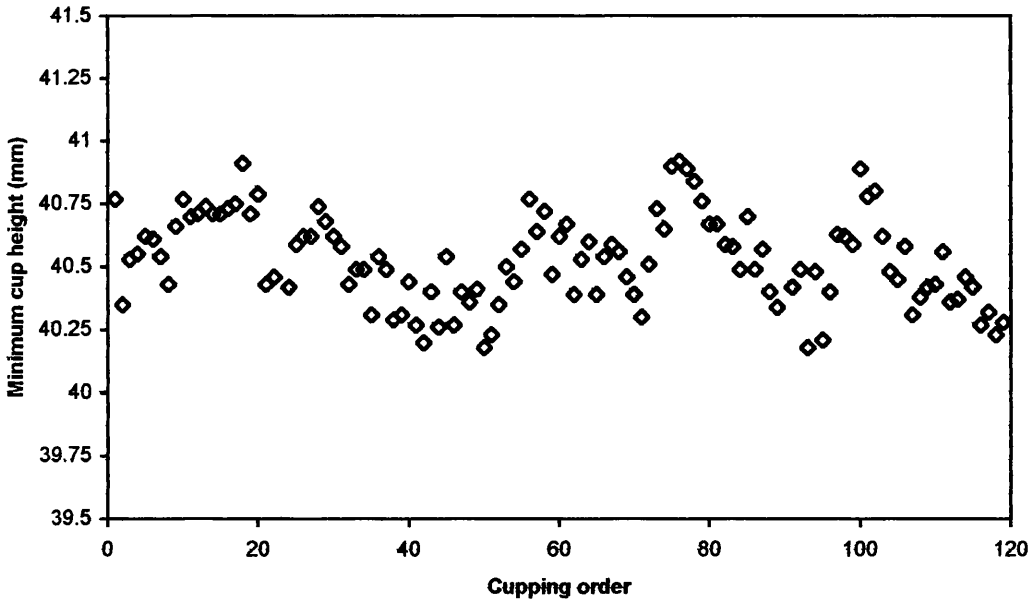

Figure 4.4. The effect of cupping order on minimum cup height for a continuous run of 120 discs.

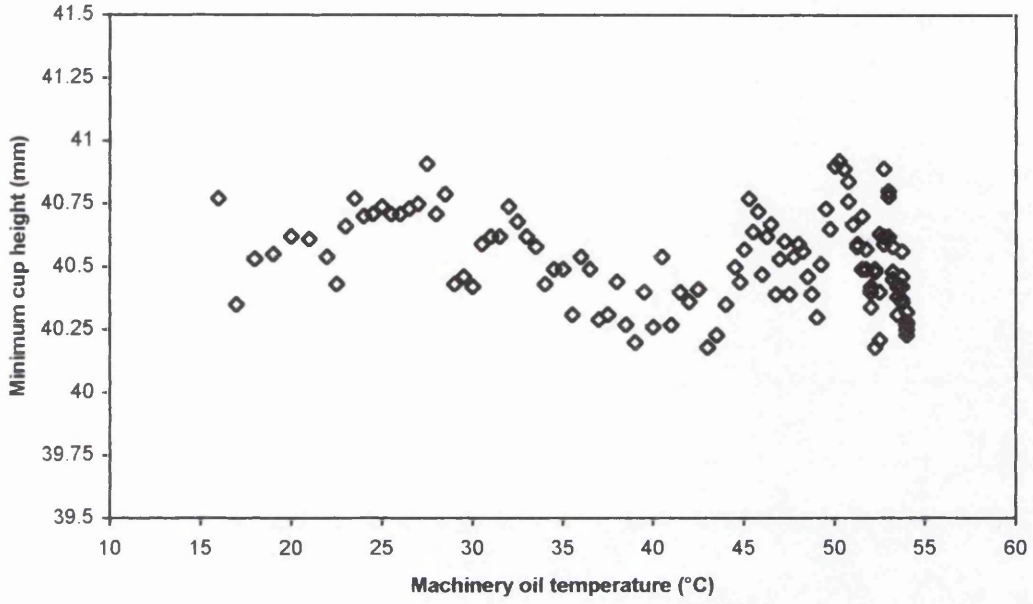

Figure 4.5. The effect of machinery oil temperature upon minimum cup height for a continuous run of 120 discs.

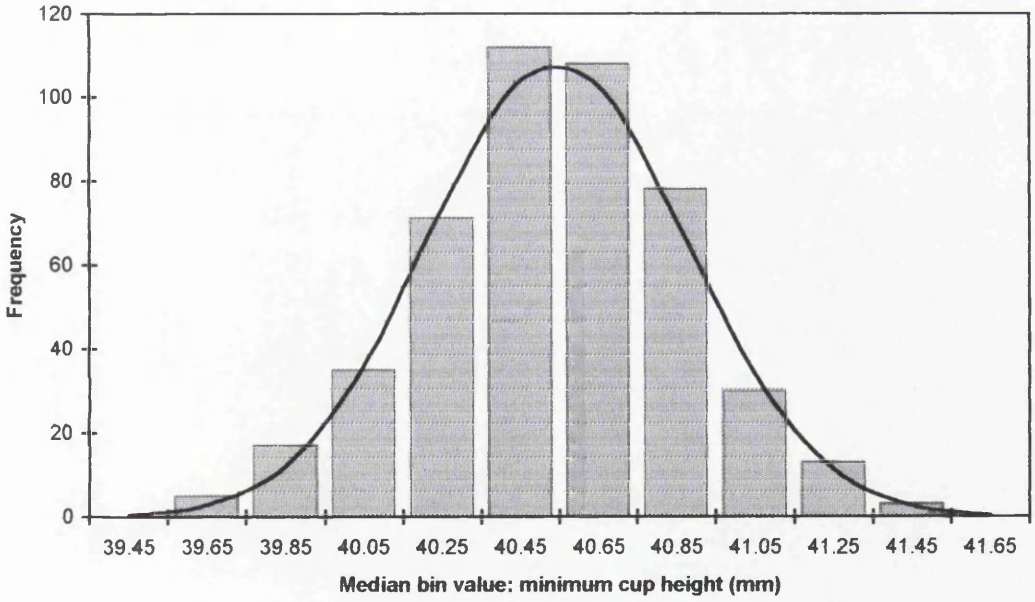

Figure 4.6. Frequency histogram of all cup ear data collected from a continuous run of 120 discs.

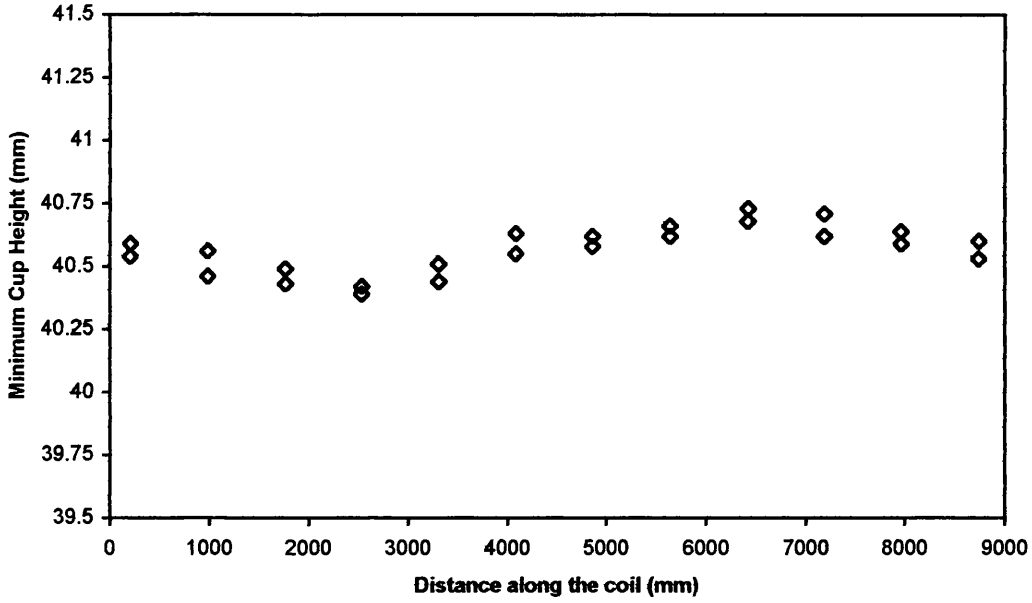

Figure 4.7. Variation in minimum cup height along the examined area.

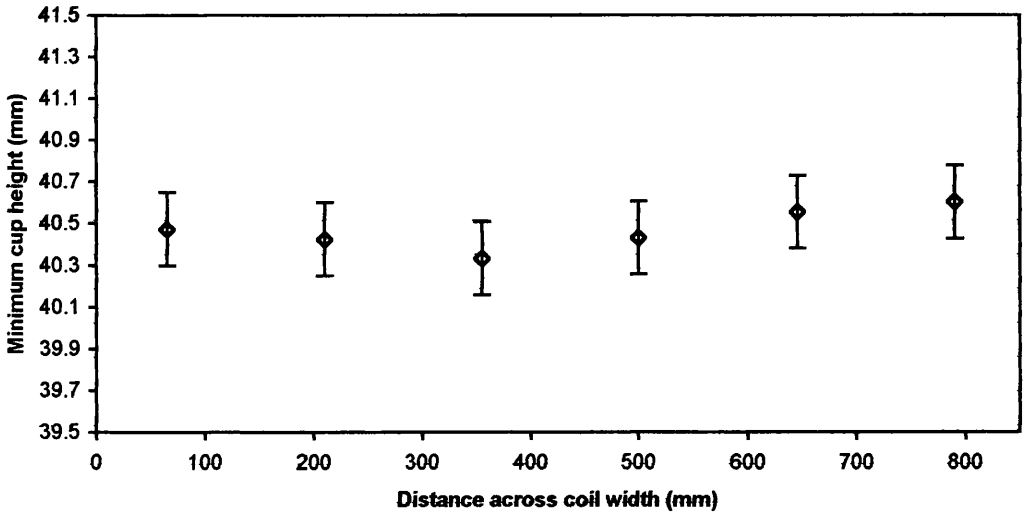

Figure 4.8. Variation in minimum cup height across the width of the examined material.

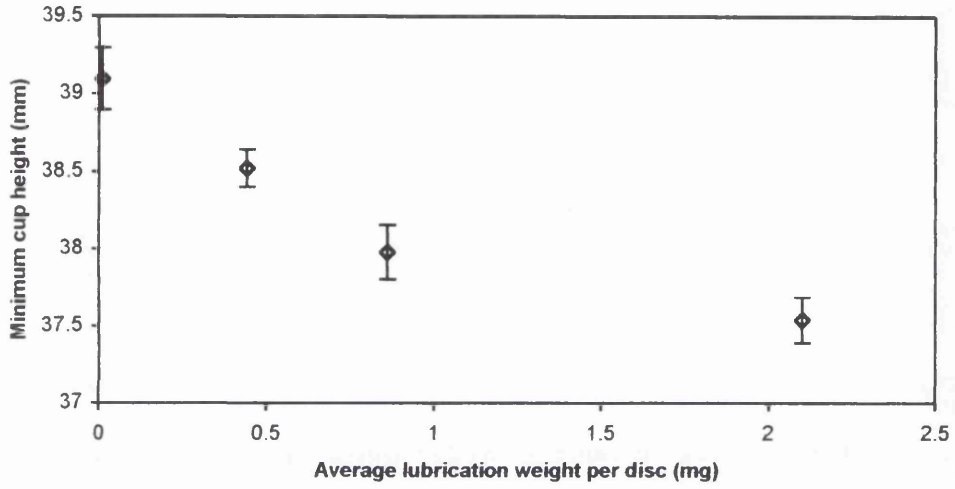

Figure 4.9. The effect of lubrication procedure on the cup height of drawn film laminated material.

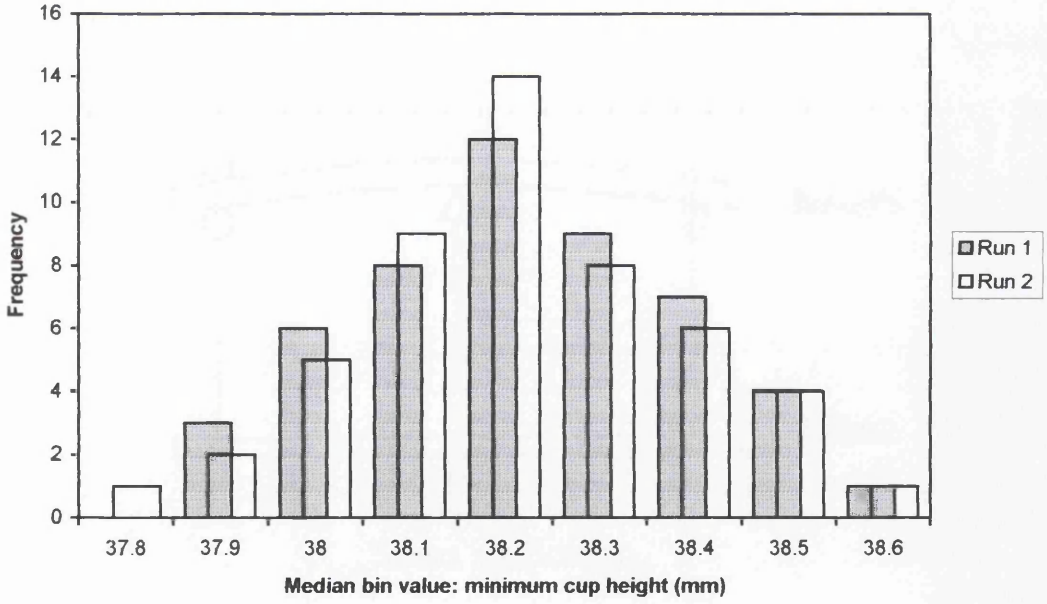

Figure 4.10. A frequency histogram of minimum cup height for two sets of 50 cups produced using the standard operating procedure for the Roell & Korthaus Cupping Press.

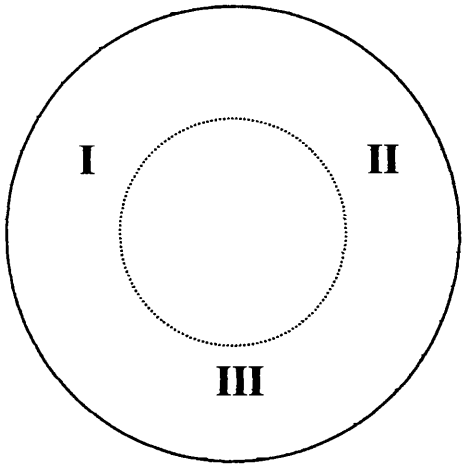

Figure 4.11. Disc sample showing nomenclature for biaxial stressing experiments.

I – Shows the date and time of the tests

II – Indicates the temperature of prestoving (if applicable)

III – Indicates the dome simulation depth (if different form standard)

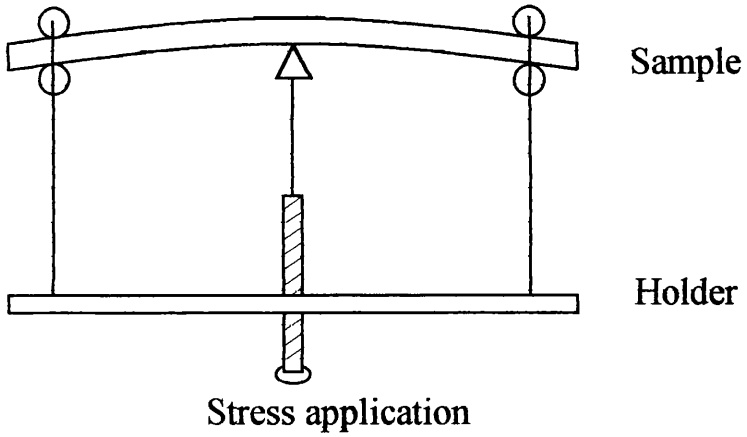

Figure 4.12. Schematic diagram of the three-point bend test commonly used in environmental stress cracking analysis.

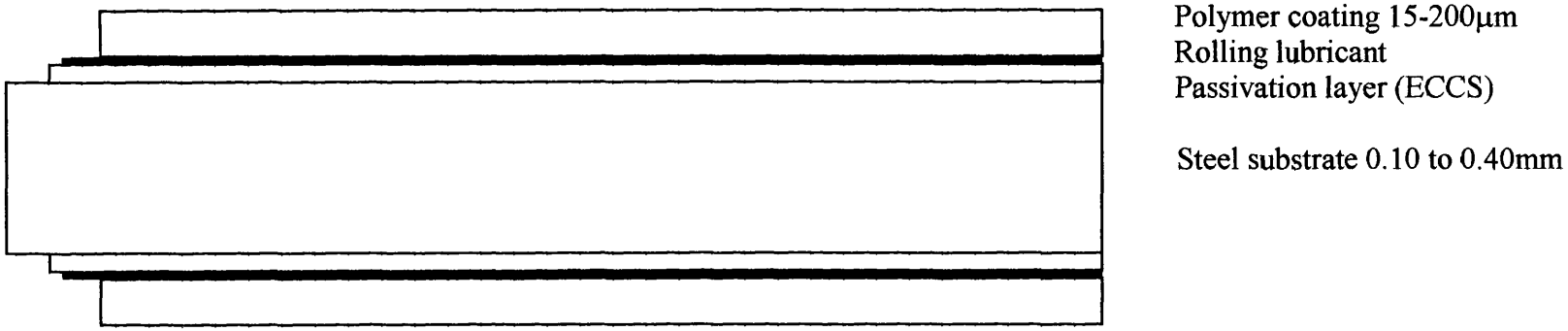

Figure 4.13. Schematic diagram showing a cross-section of a typical film laminate material

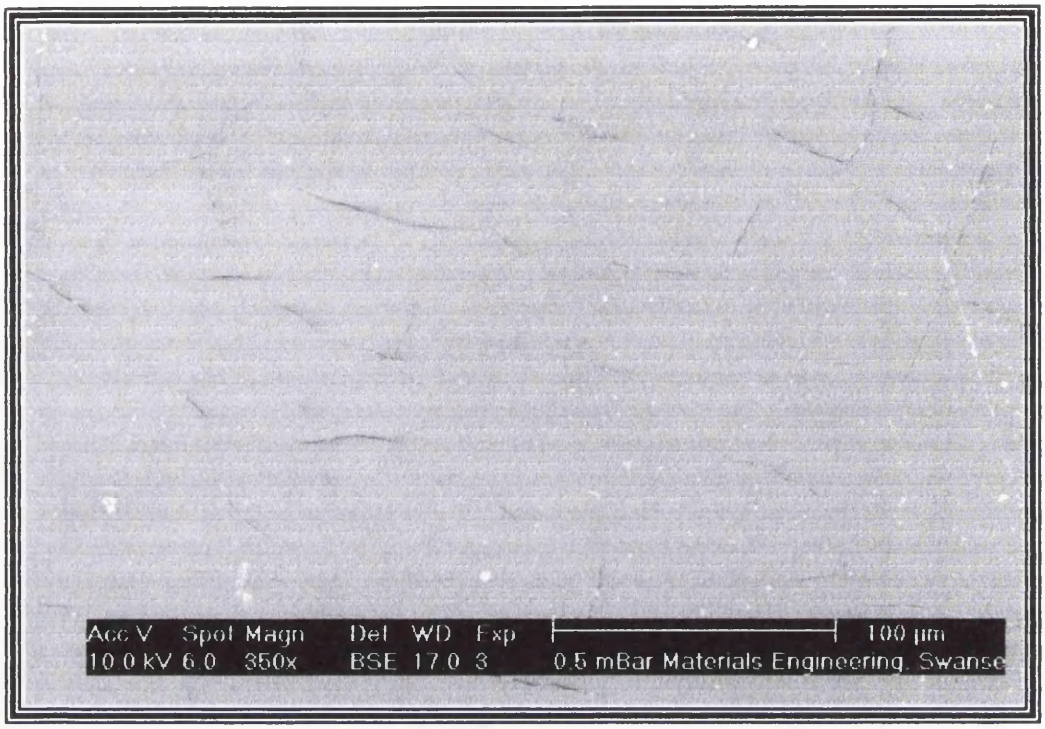

Figure 4.14. Scanning electron microscopy analysis of crazing captured using the low pressure apparatus at University of Wales Swansea.

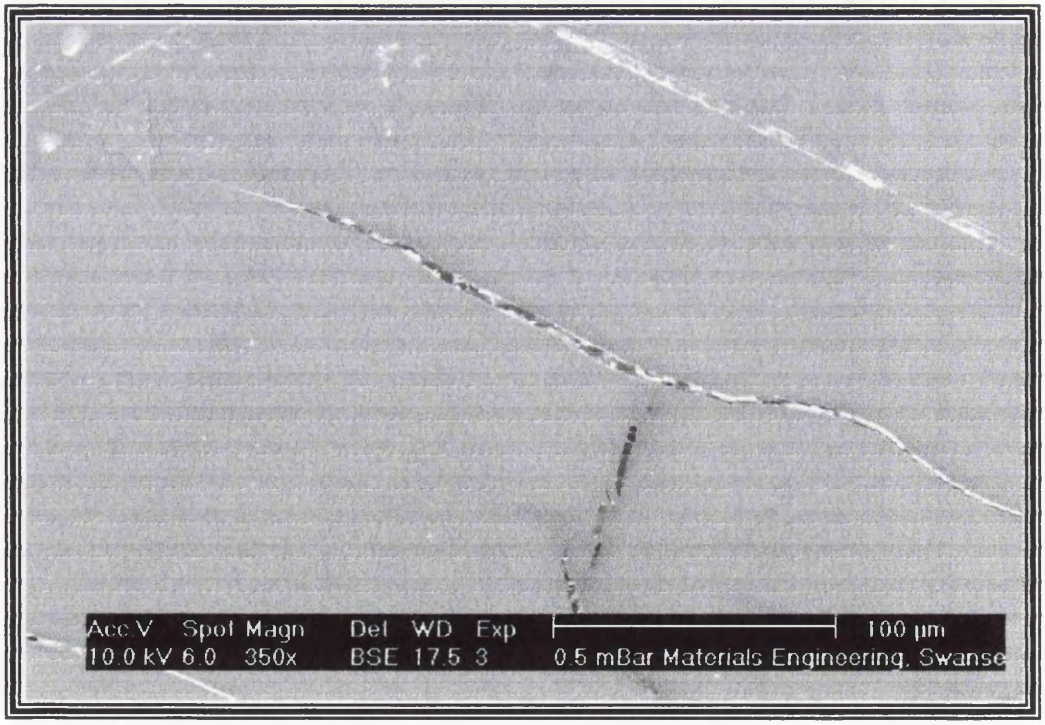

Figure 4.15. Scanning electron microscopy analysis of crazing captured using the low pressure apparatus at University of Wales Swansea, taken approximately 2mm from Figure 4.14.

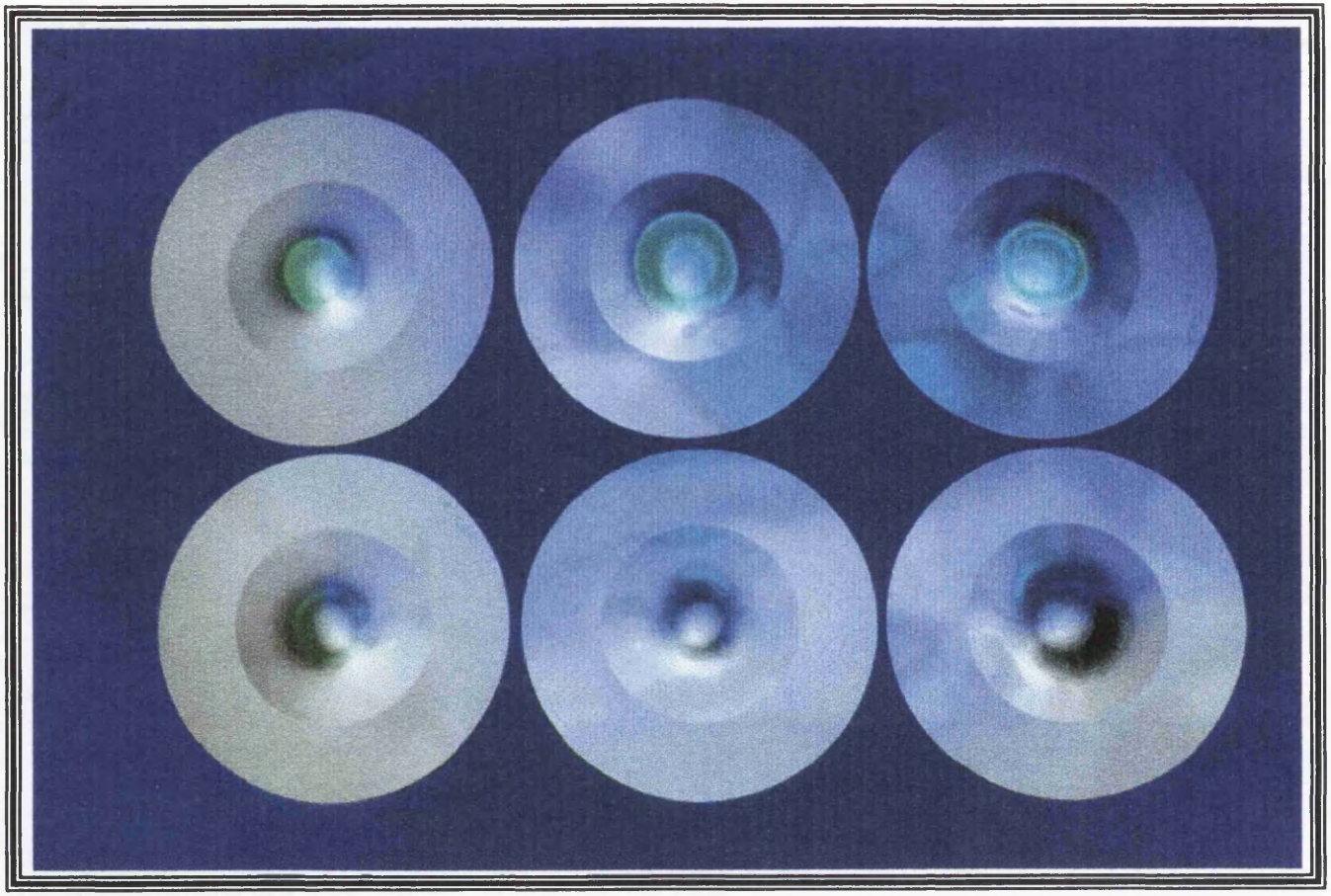

Figure 4.16. Photographic representation of the range of crazing observed in film laminated materials, captured using ultraviolet inks and light.

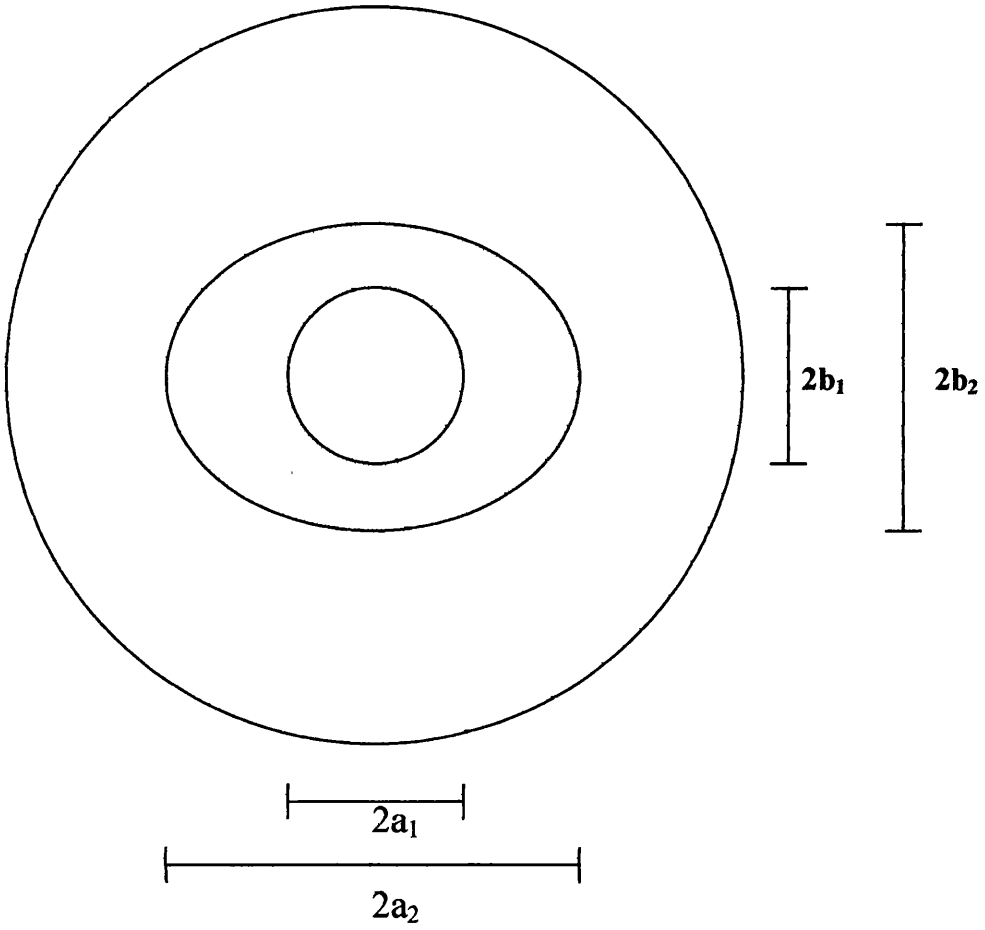

Figure 4.17. Schematic diagram representing a biaxially stressed disc and the critical regions used for measurement to establish the total crazed area.

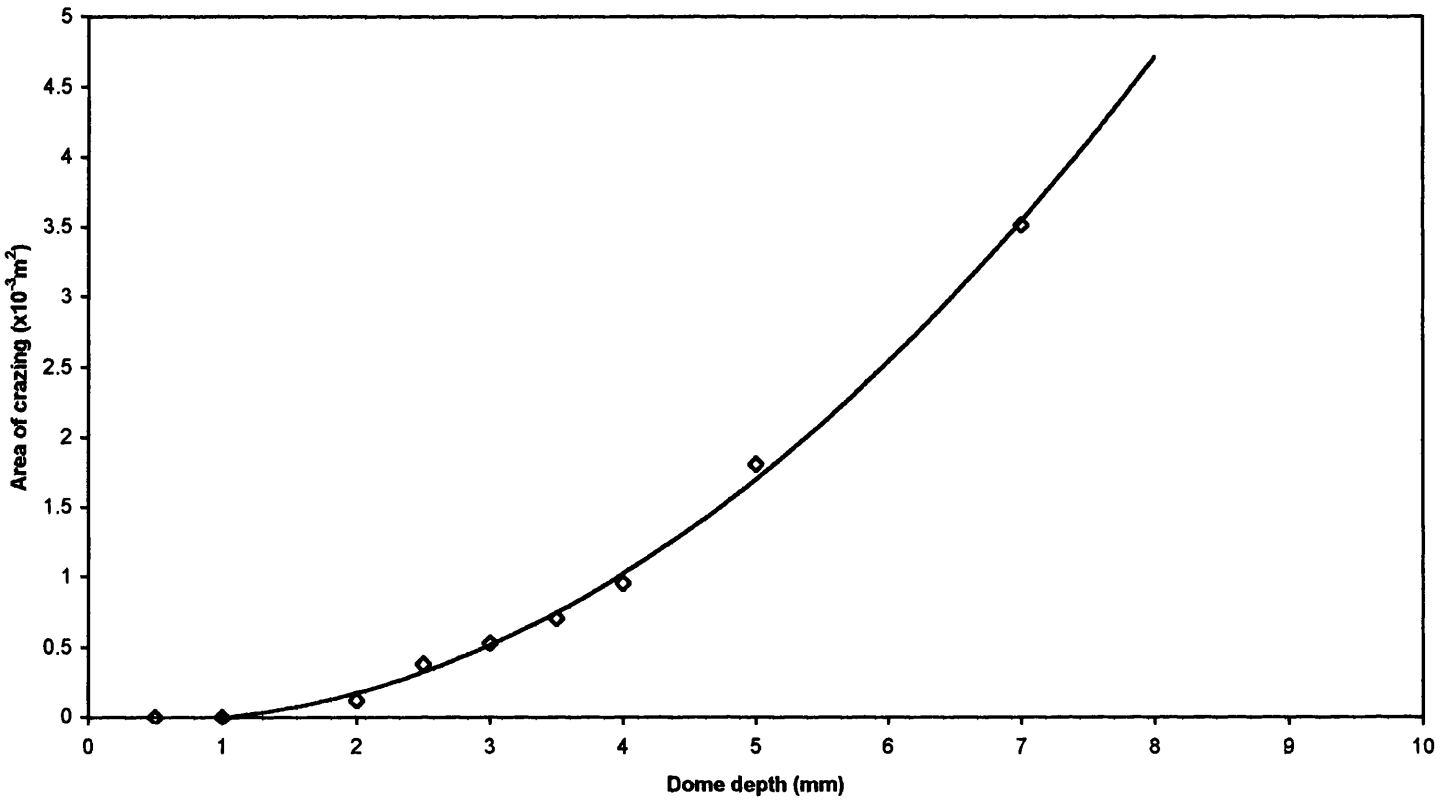

Figure 4.18. The effect of dome depth on area of crazing for a typical film laminated material.

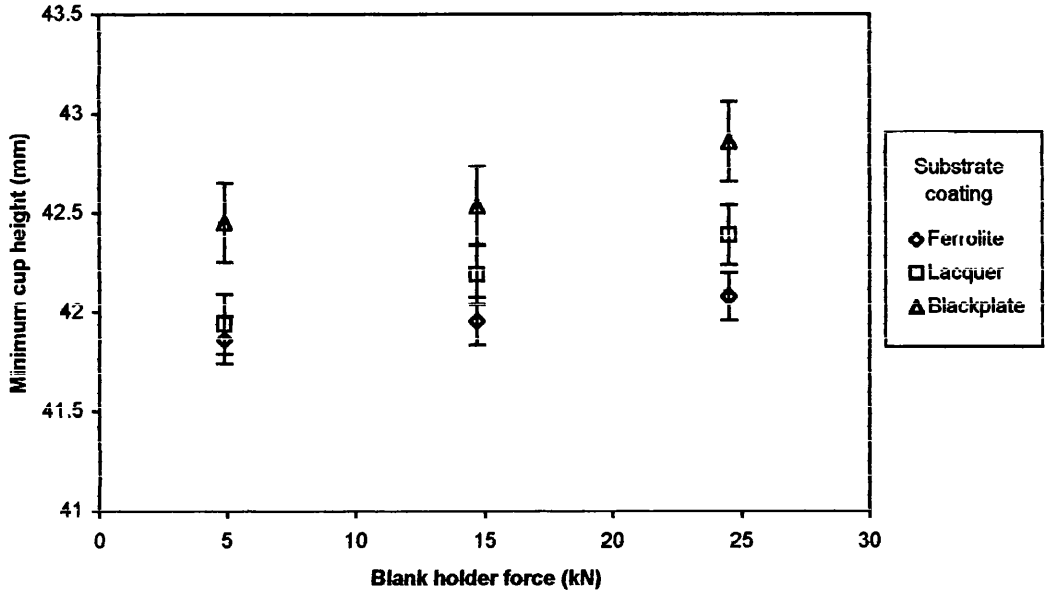

Figure 5.1. Comparison of different substrate coating types on the minimum cup height of material with 0.195mm nominal gauge.

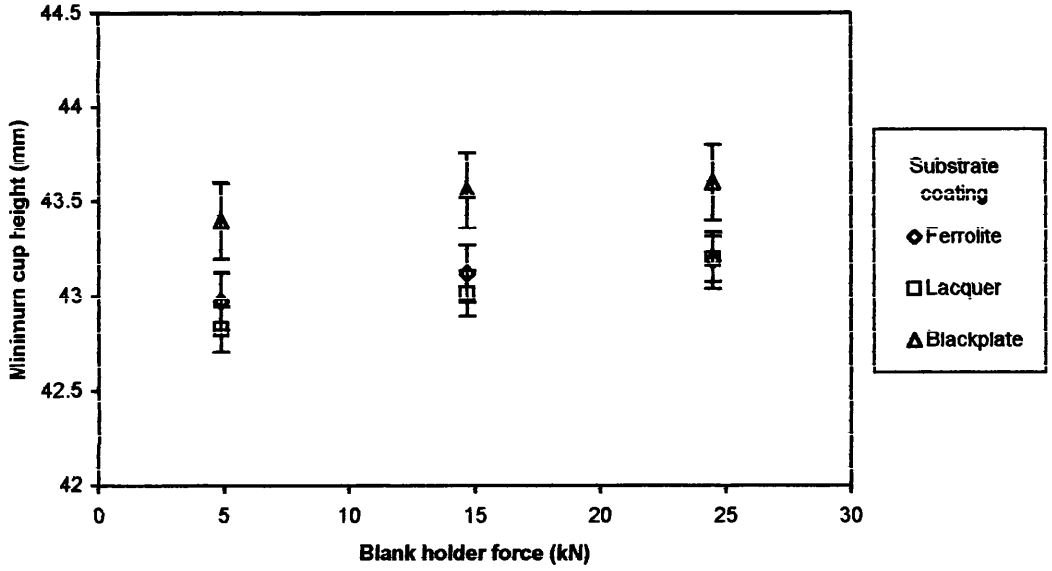

Figure 5.2. Comparison of different substrate coating types on the minimum cup height of material with 0.26mm nominal gauge.

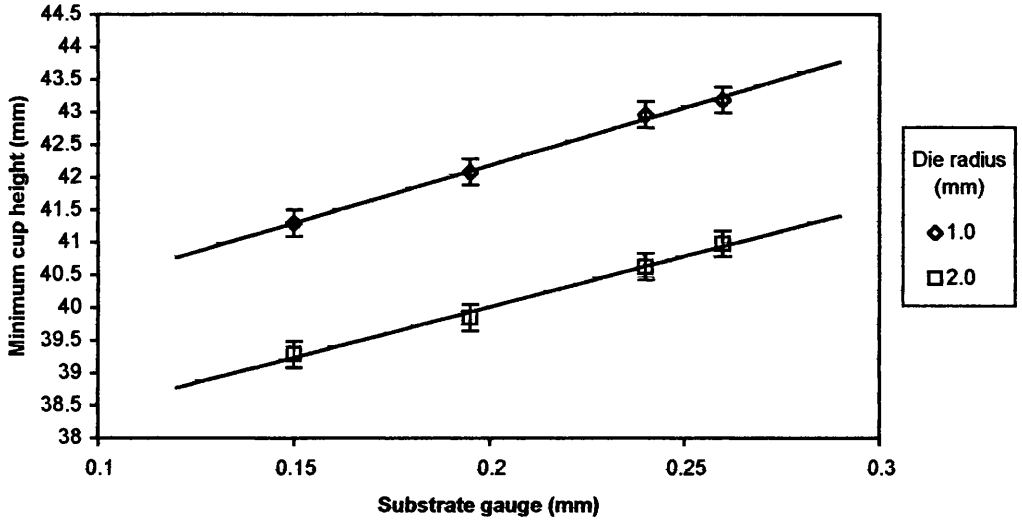

Figure 5.3. Effect of substrate gauge on minimum cup height for film laminated materials of 620MPa nominal ultimate tensile strength.

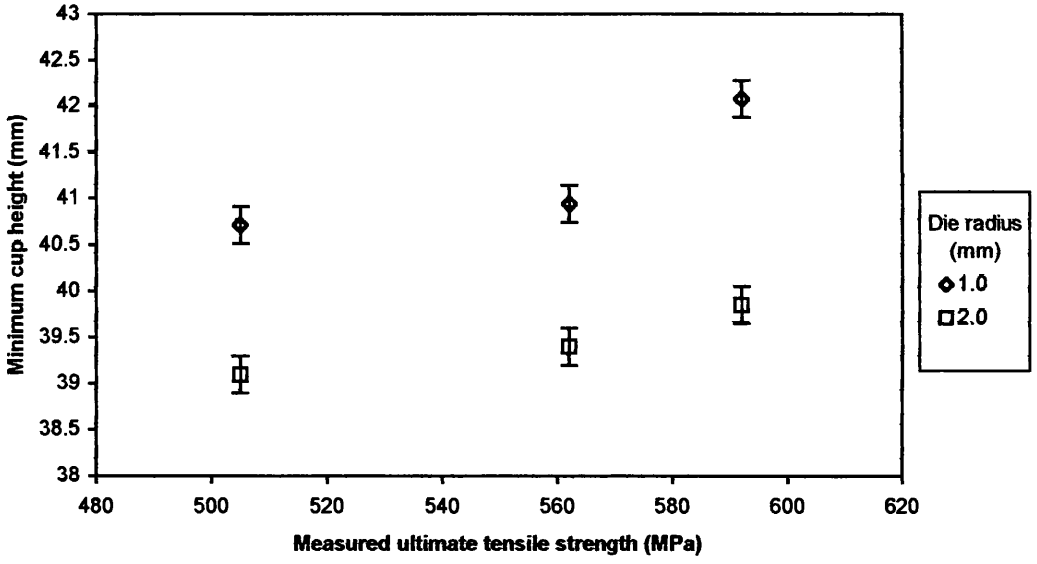

Figure 5.4. The effect of substrate strength on minimum cup height for film laminated materials of 0.19mm nominal thickness.

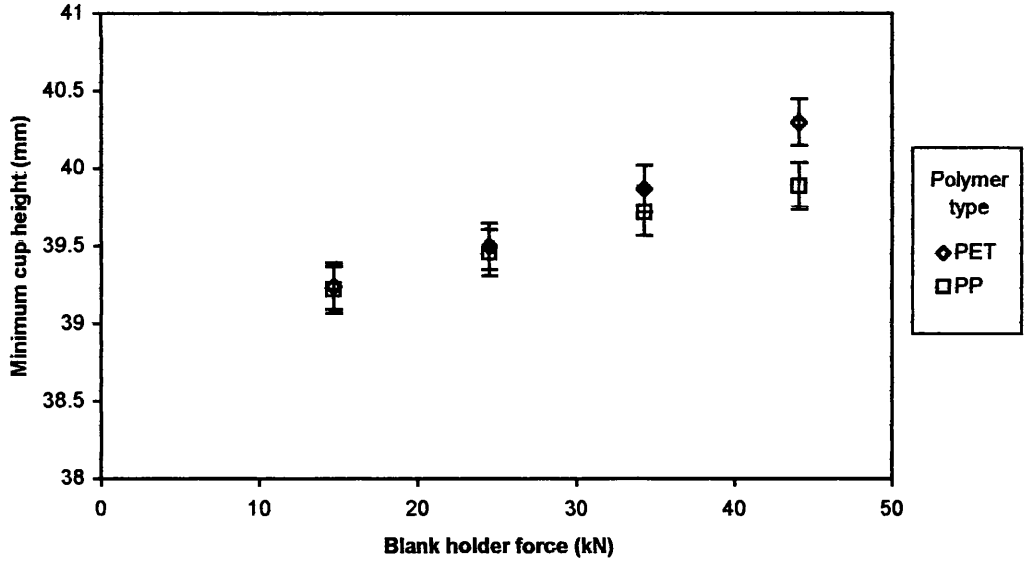

Figure 5.5. The effect of polymer coating type on the minimum height of film laminated materials for cups produced over a 1mm die radius.

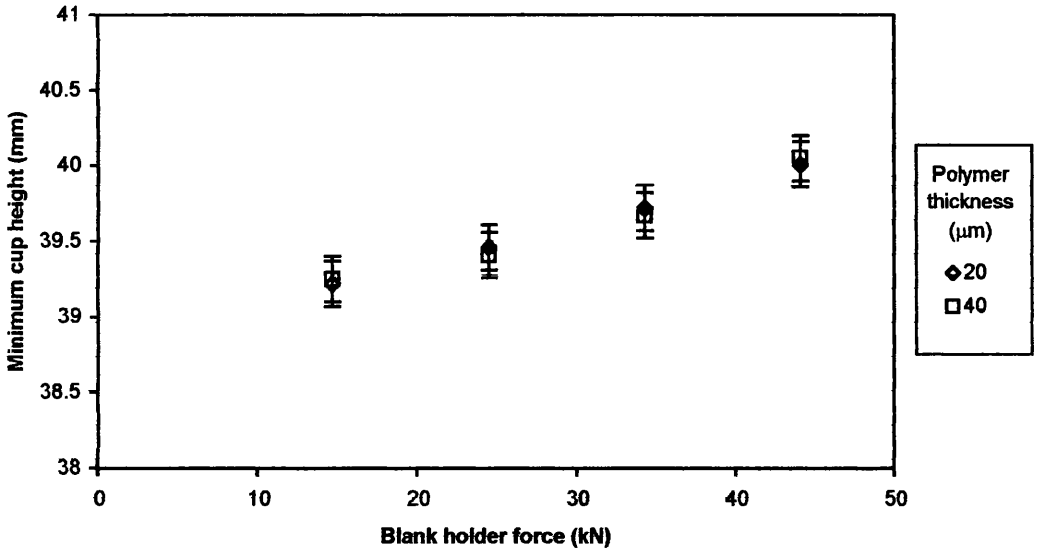

Figure 5.6. The effect of polypropylene coating thickness on the minimum cup height of film laminated material produced over a 1mm die radius.

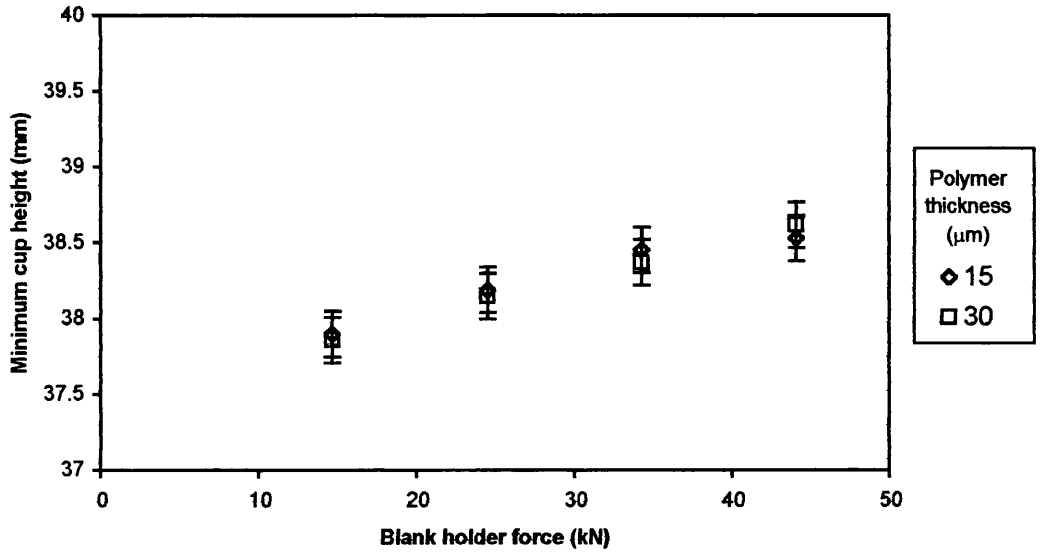

Figure 5.7. The effect of polyethylene terephthalate coating thickness on the minimum cup height of film laminated material produced over a 1mm die radius.

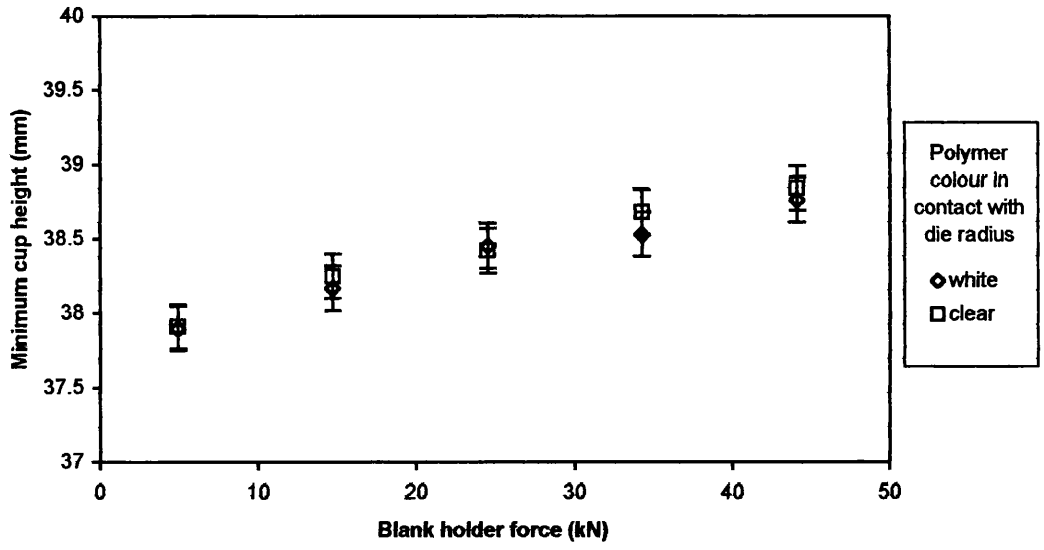

Figure 5.8. The effect of polyethylene terephthalate pigmentation on the cup height of film laminated materials.

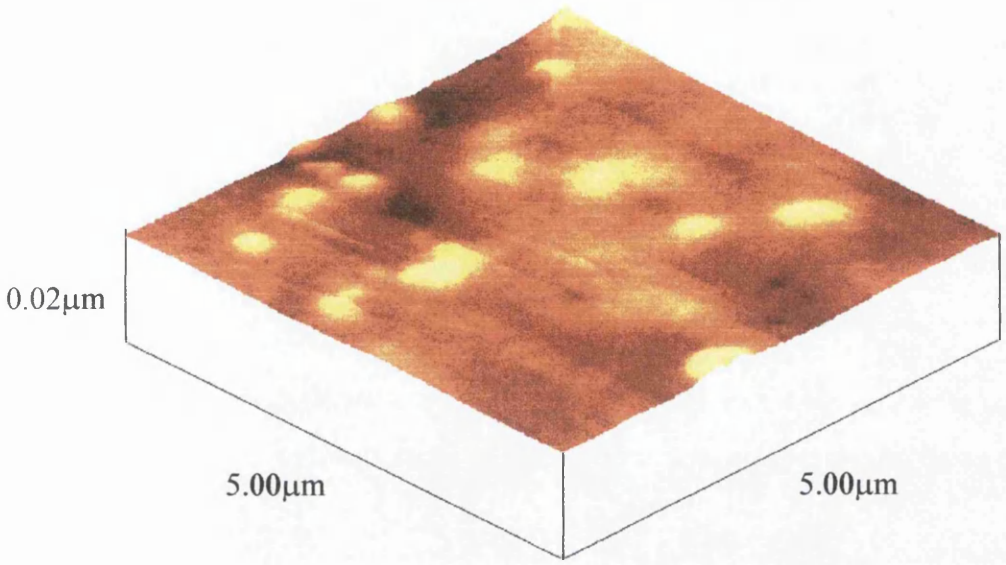

Figure 5.9. AFM imaging of the white surface of a polyethylene terephthalate film laminated material.

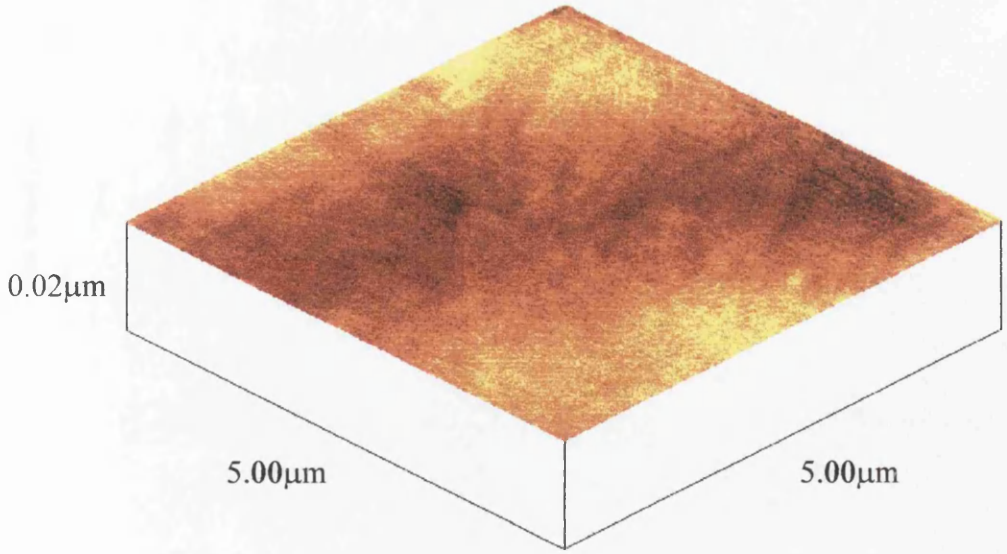

Figure 5.10. AFM imaging of the clear surface of a polyethylene terephthalate film laminated material.

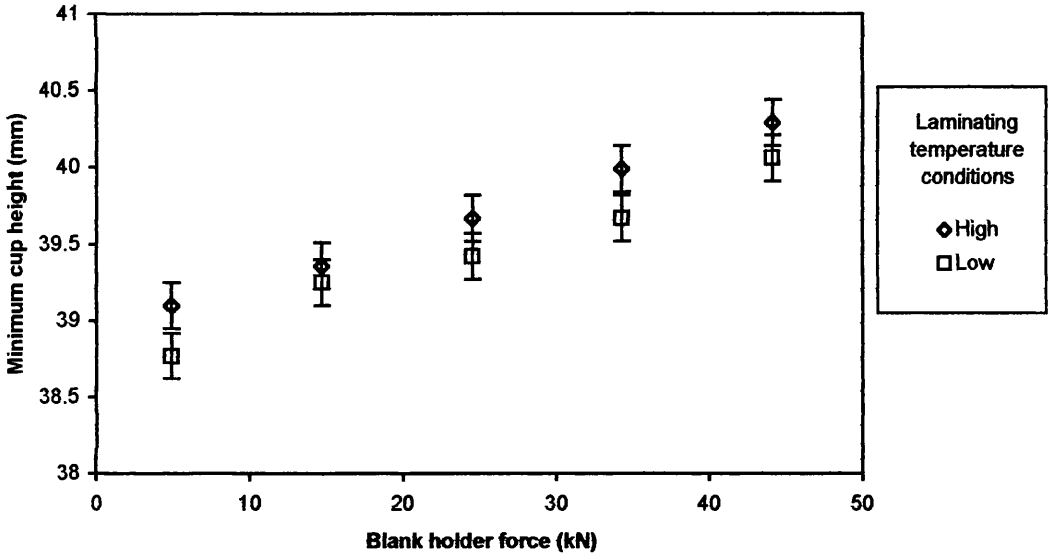

Figure 5.11. The effect of polypropylene laminating conditions on the minimum cup height of film laminated material produced over a 1mm die radius.

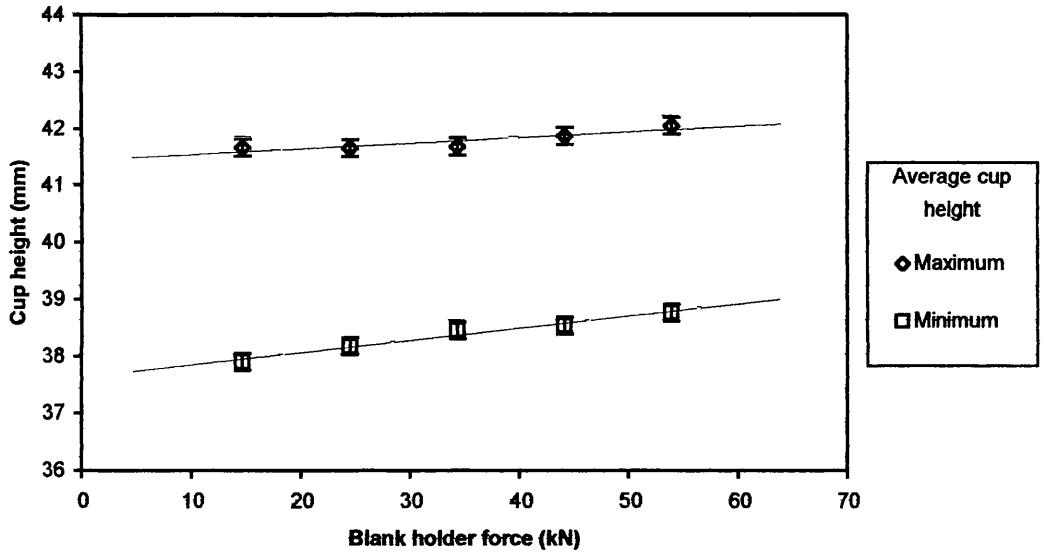

Figure 5.12. The effect of blank holder force on the average minimum and average maximum cup heights of film laminated material.

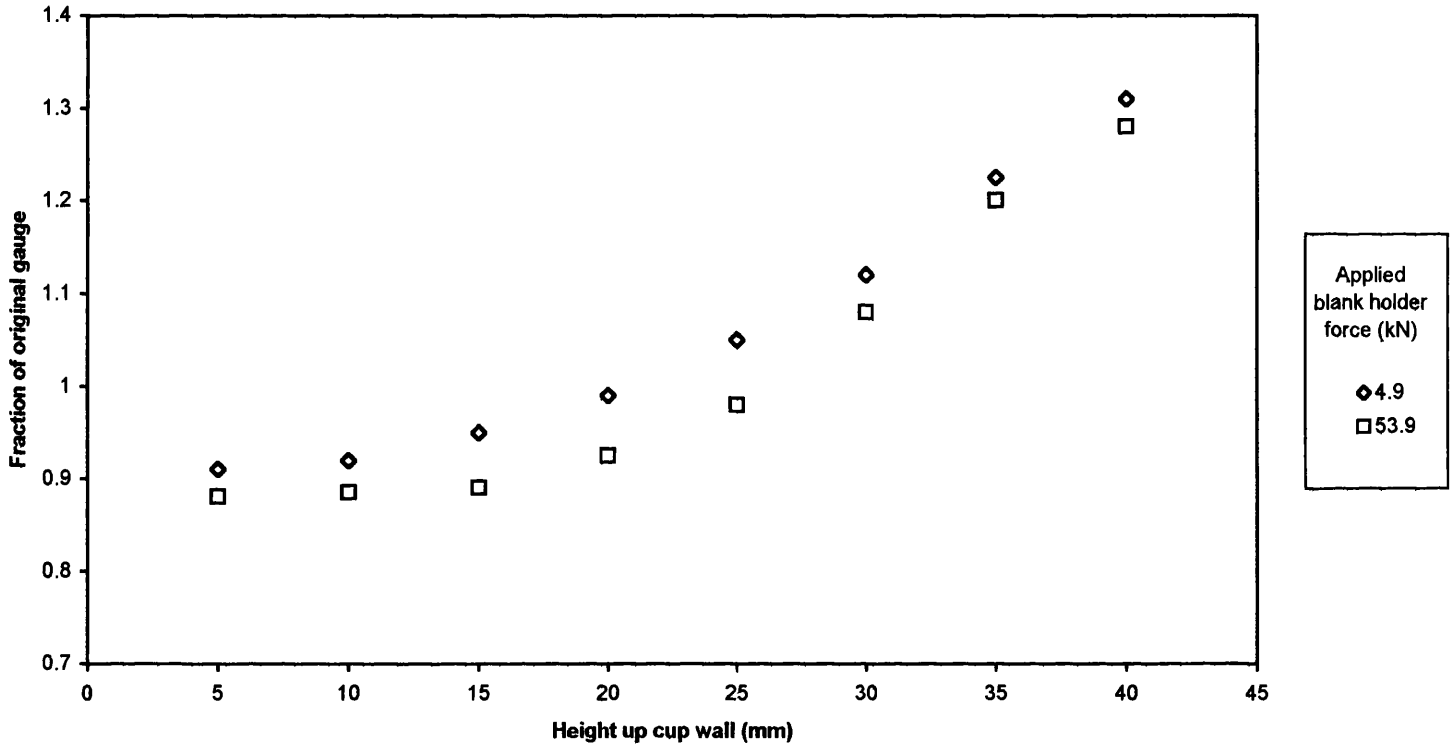

Figure 5.13. Wall profiles of cups produced at blank holder forces at the extremes of the operating window using a 1.36mm die radius.

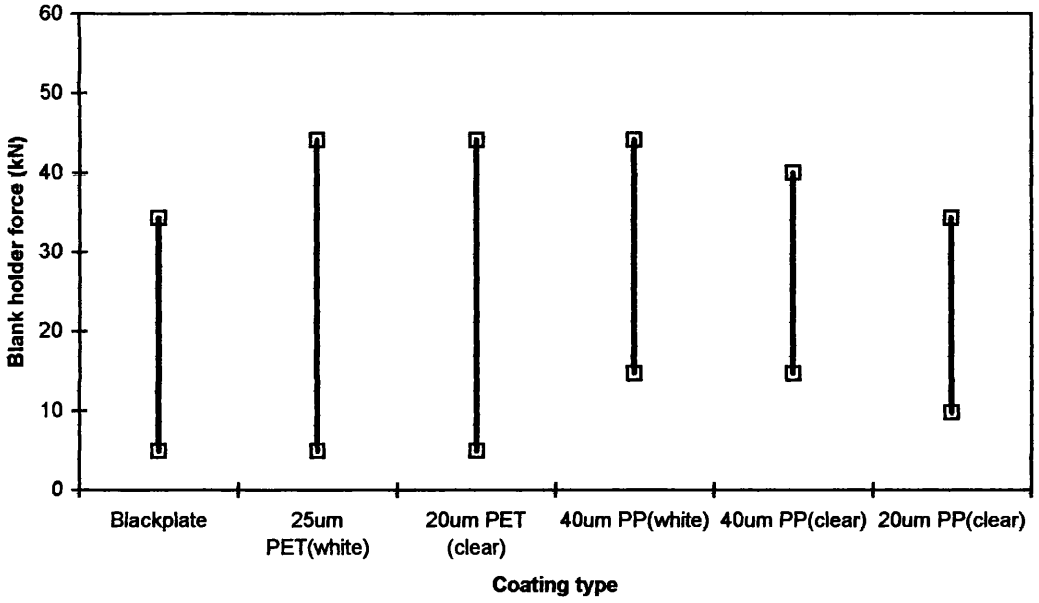

Figure 5.14. The operating window of blank holder forces for production of a successful cup for a range of polymer coatings using a 1mm die radius.

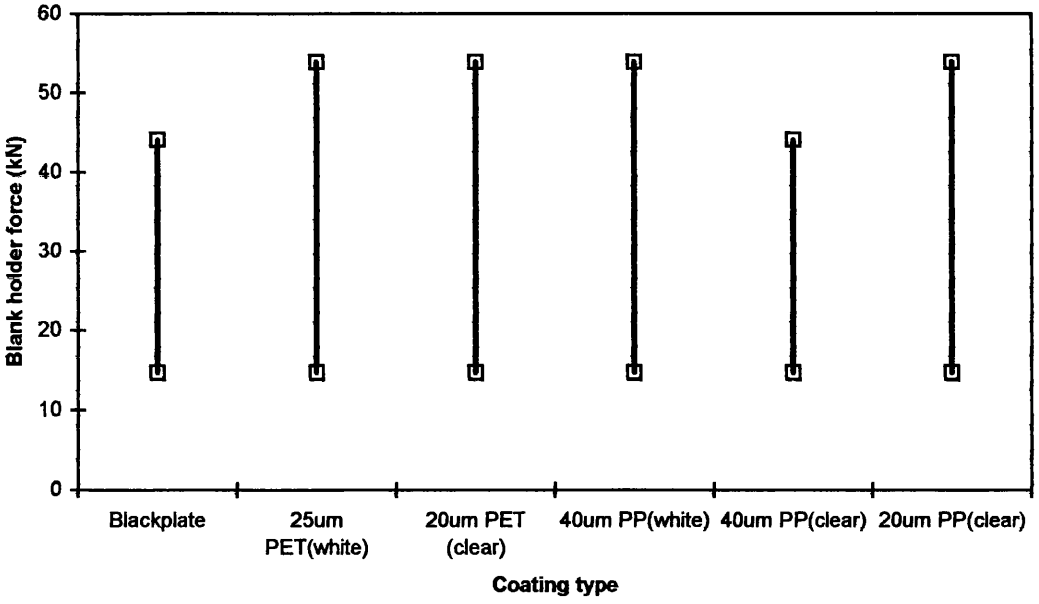

Figure 5.15. The operating window of blank holder forces for production of a successful cup for a range of polymer coatings using a 2mm die radius.

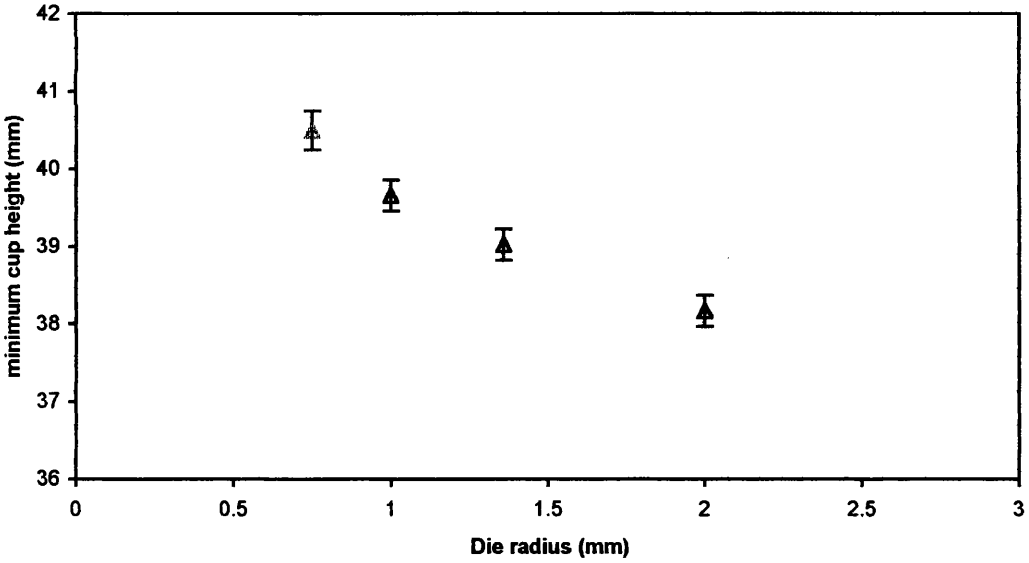

Figure 5.16. The effect of die radius on cup height of film laminated materials generated from a cut edge of 125mm.

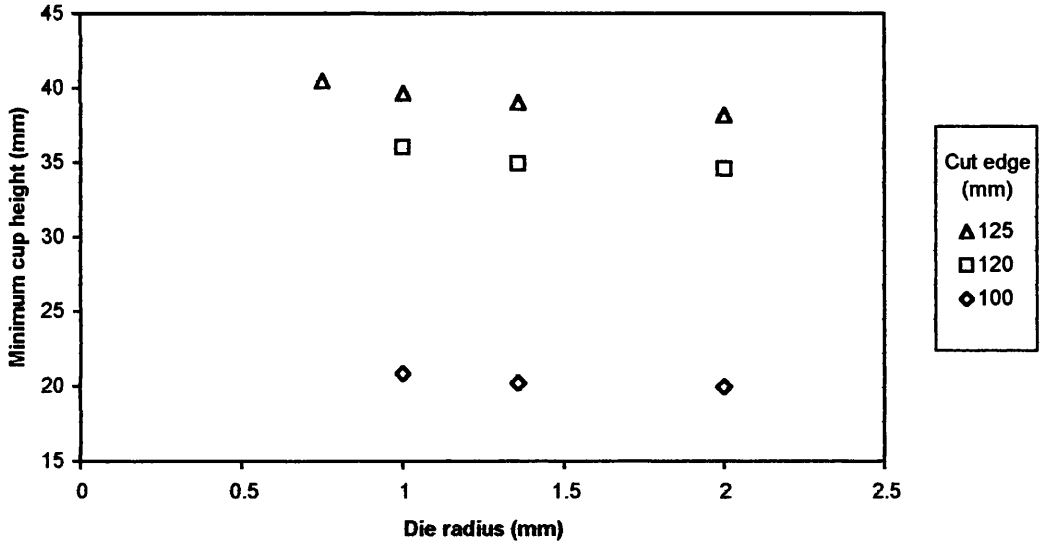

Figure 5.17. The effect of die radius on cup height of film laminated materials generated from a range of cut edge dimensions.

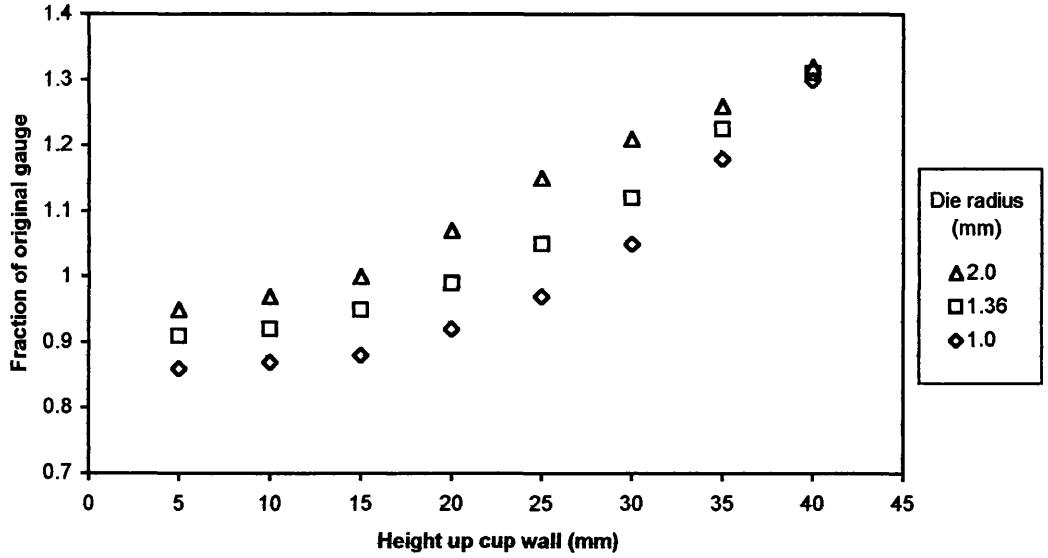

Figure 5.18. Wall profiles of cups produced over a range of die radii from a cut edge of 125mm.

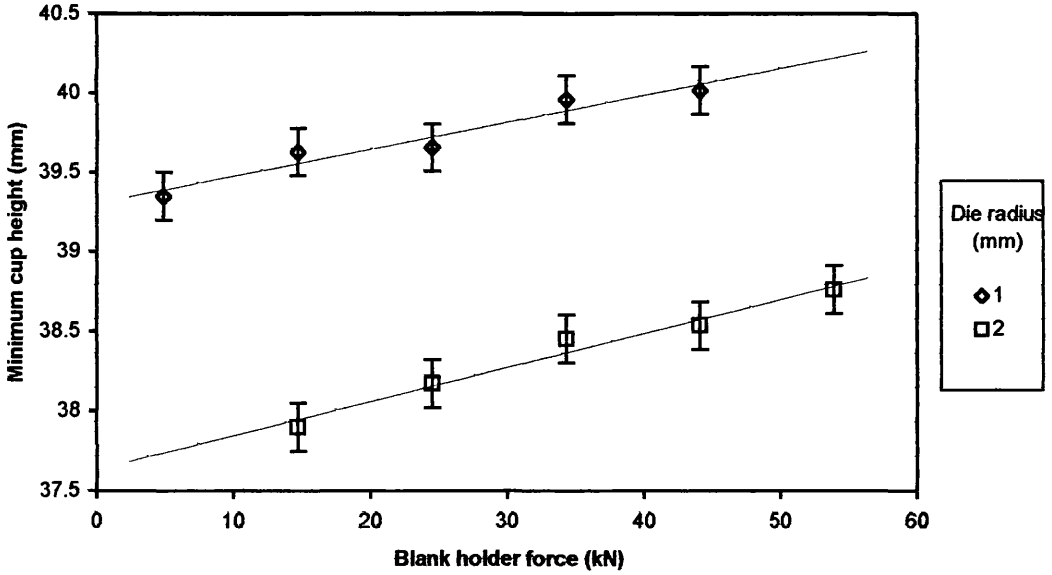

Figure 5.19. The effect of die radius and blank holder force on the minimum cup height of film laminated material.

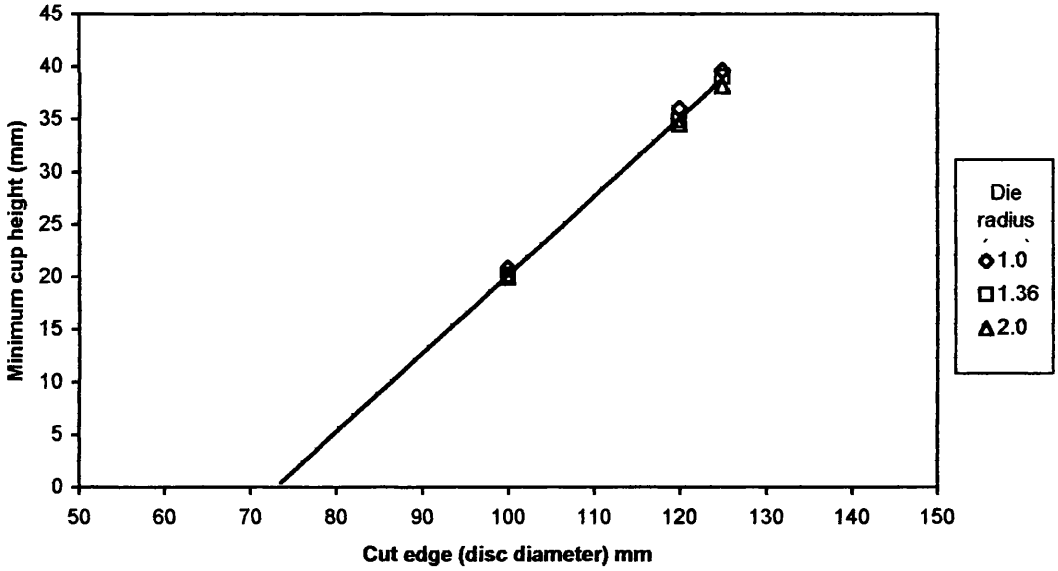

Figure 5.20. The effect of die radius and cut edge dimensions on the minimum cup height of film laminated material.

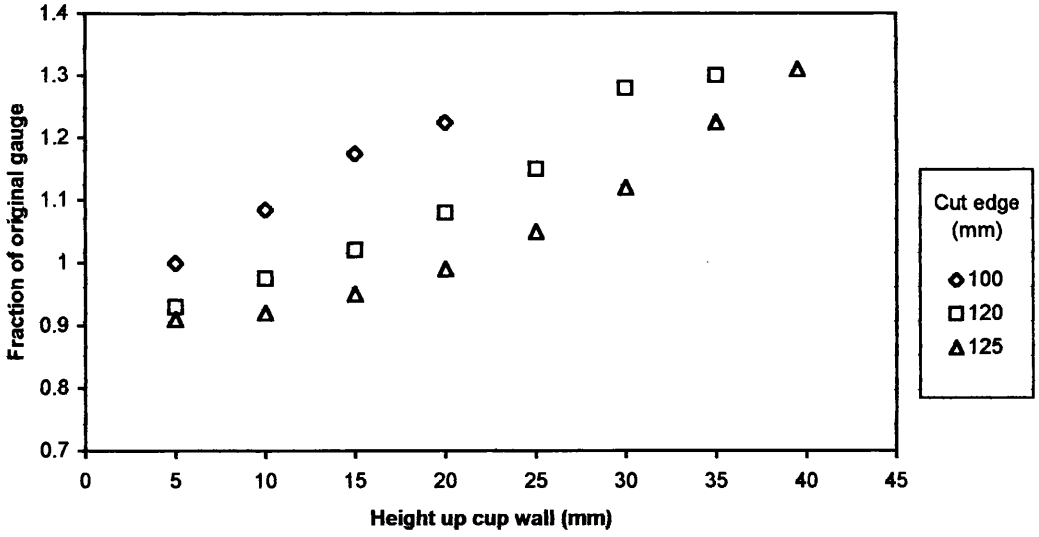

Figure 5.21. The effect of cut edge dimensions on the wall profile of film laminated material produced over a 1.36mm die radius.

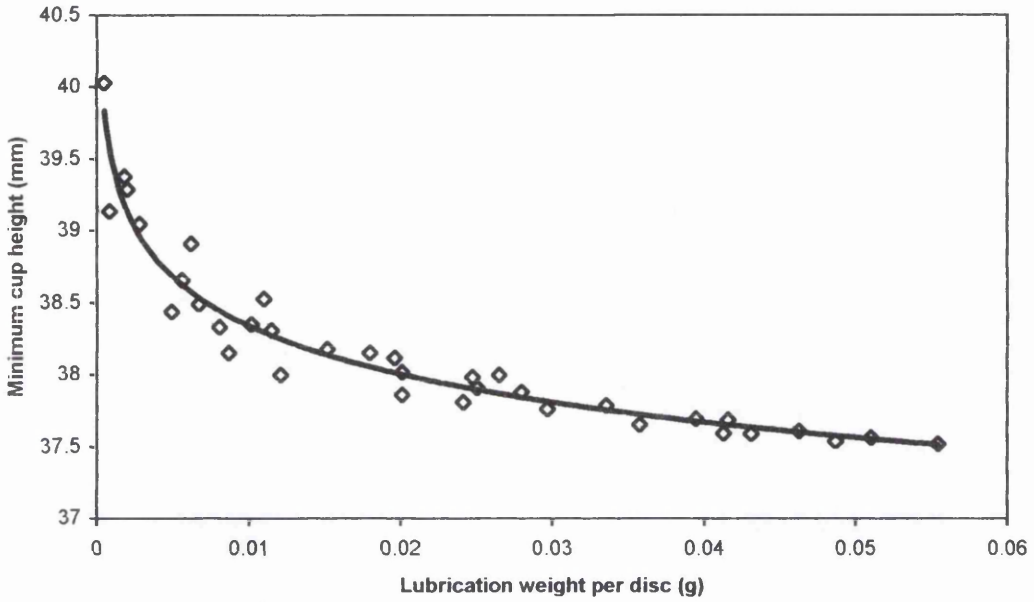

Figure 5.23. The effect of lubrication weight upon the cup height of centre only samples taken from film laminated material.

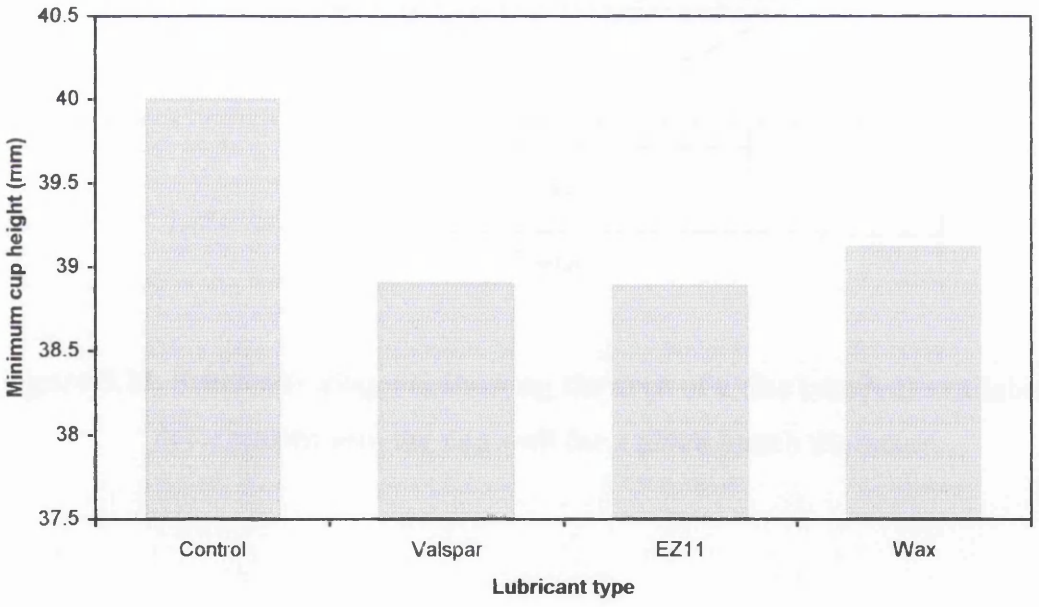

Figure 5.24. Comparison of average minimum cup height produced from identical material, but with differing lubrication types.

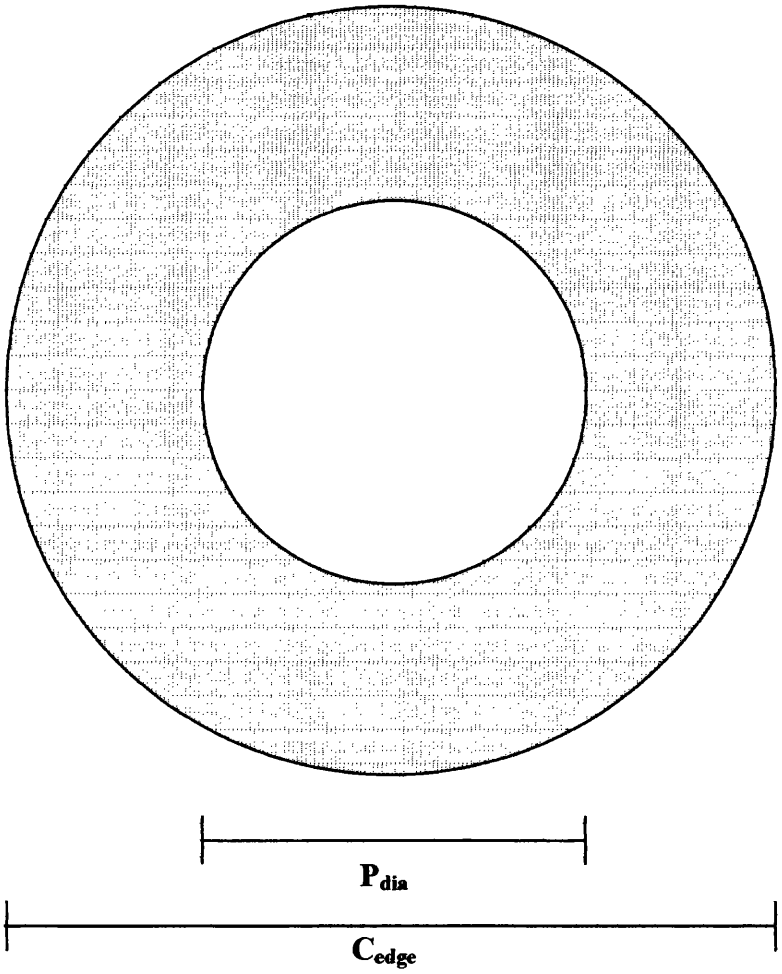

Figure 5.25. Schematic diagram showing the area of a disc (shaded) available for deformation into the cup wall for a given punch diameter.

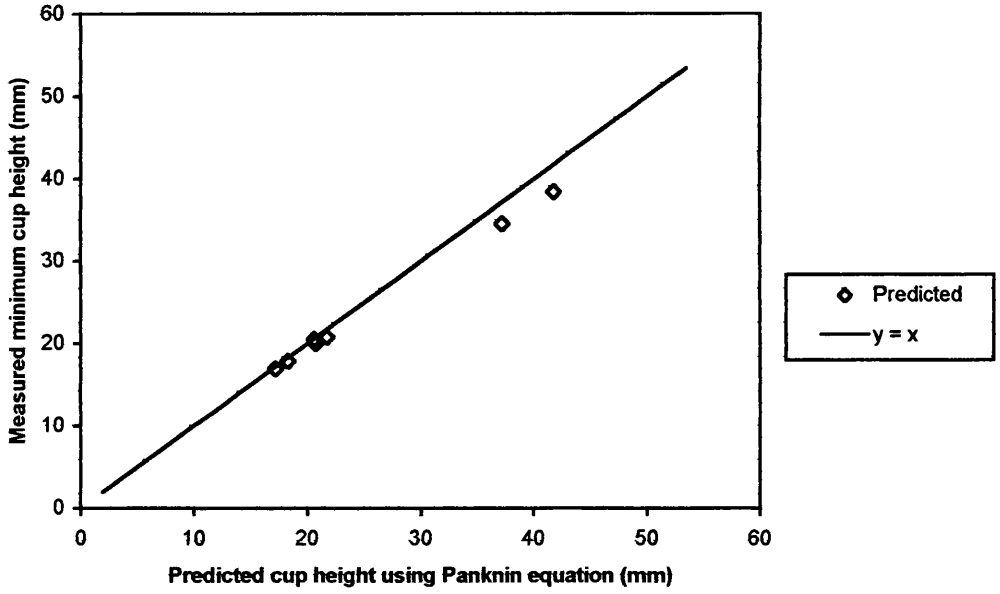

Figure 5.26. Actual and predicted cup height using Panknin equation for a typical film laminated material.

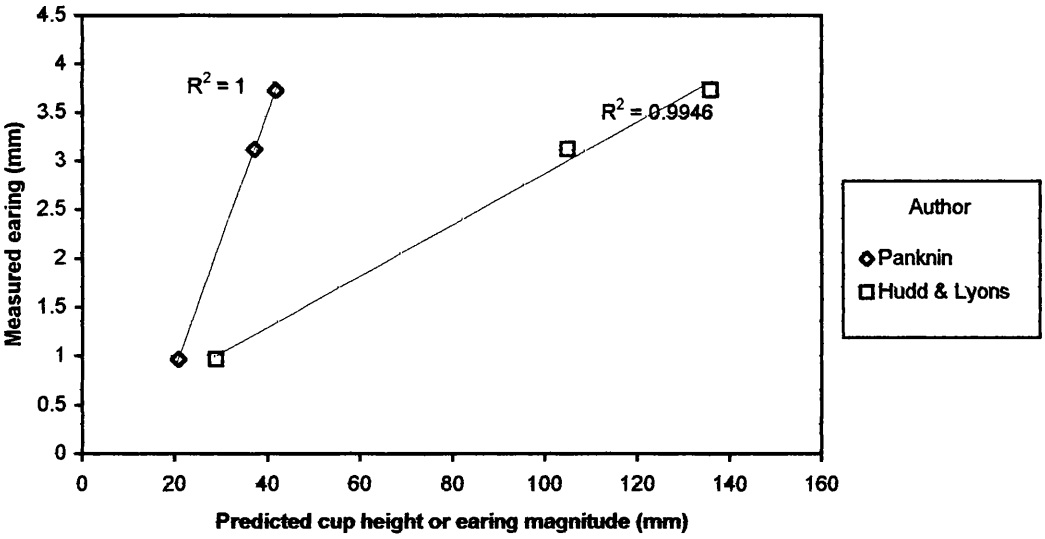

Figure 5.27. Measured earing levels of film laminated material compared to predicted values.

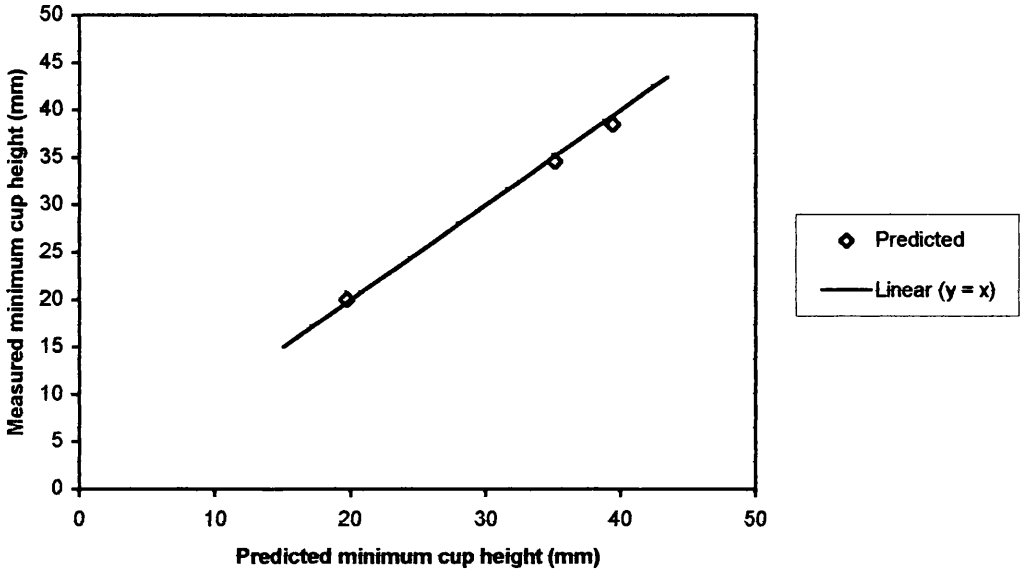

Figure 5.28. Measured and predicted cup height of a film laminated material using the developed equation, which considers earing and punch nose radius (for 66.79mm punch diameter).

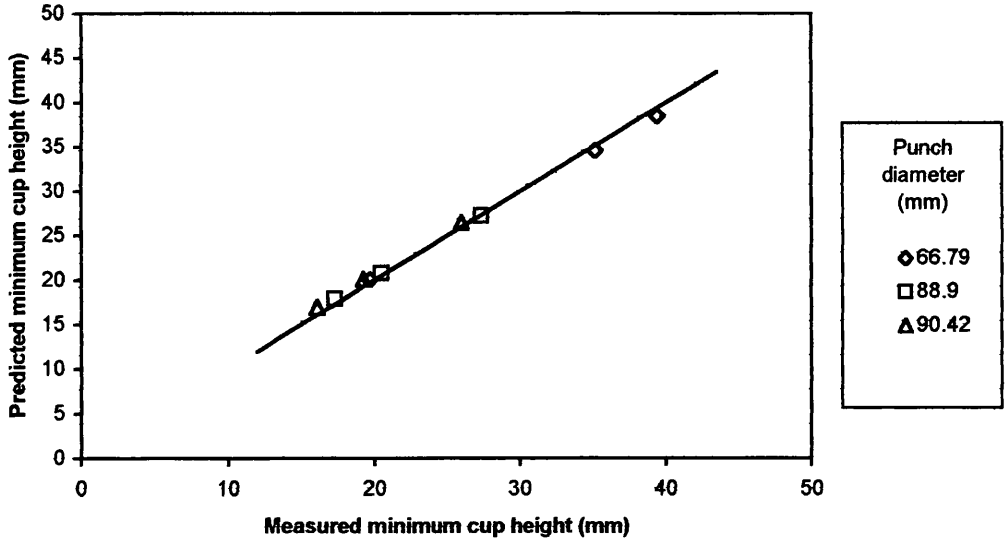

Figure 5.29. Measured and predicted cup height of a film laminated material using the developed equation, which considers earing and punchnose radius (for all punch diameters considered).

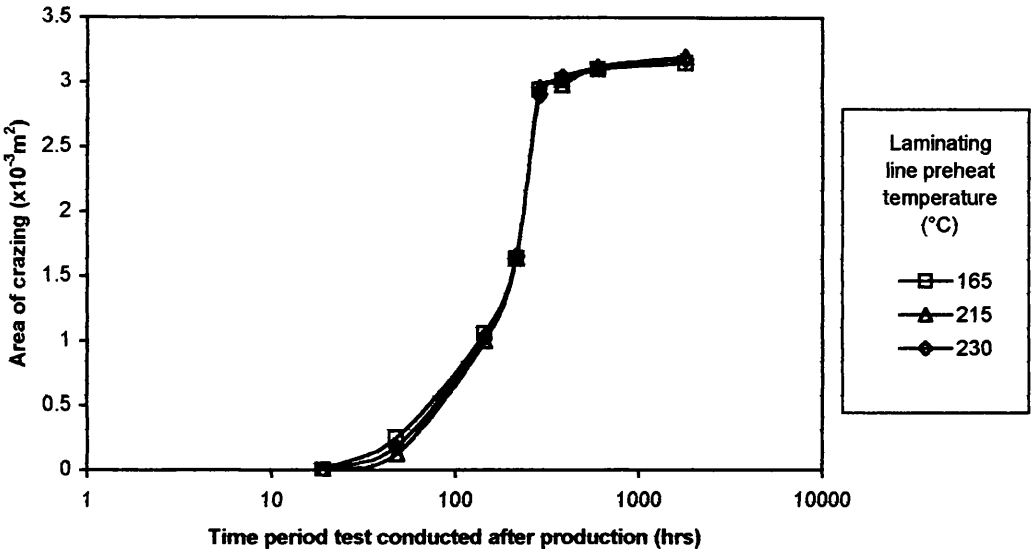

Figure 6.1. The influence of laminating line preheat temperature on the craze susceptibility of recently produced film laminated material.

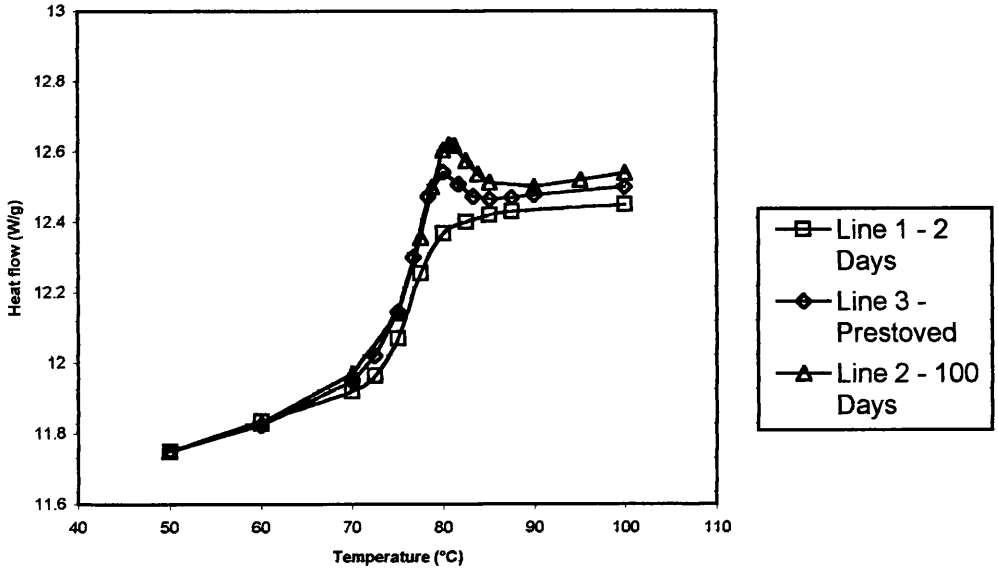

Figure 6.2. Partial differential scanning calorimetry plot of a typical film laminated coating at a range of time periods after production.

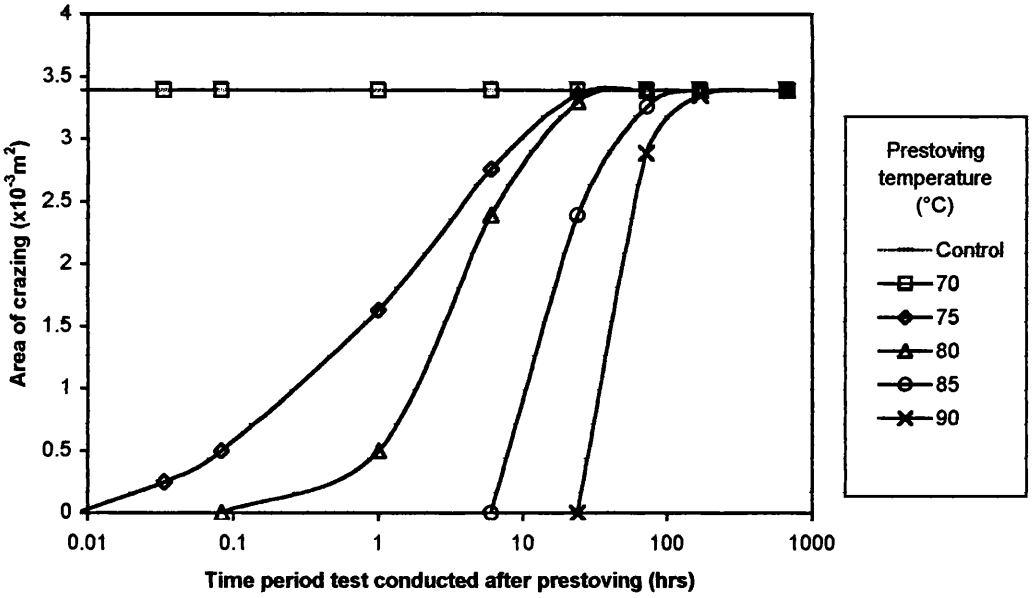

Figure 6.3. The effect of prestoving temperature on the crazed area of biaxially stressed film laminated material.

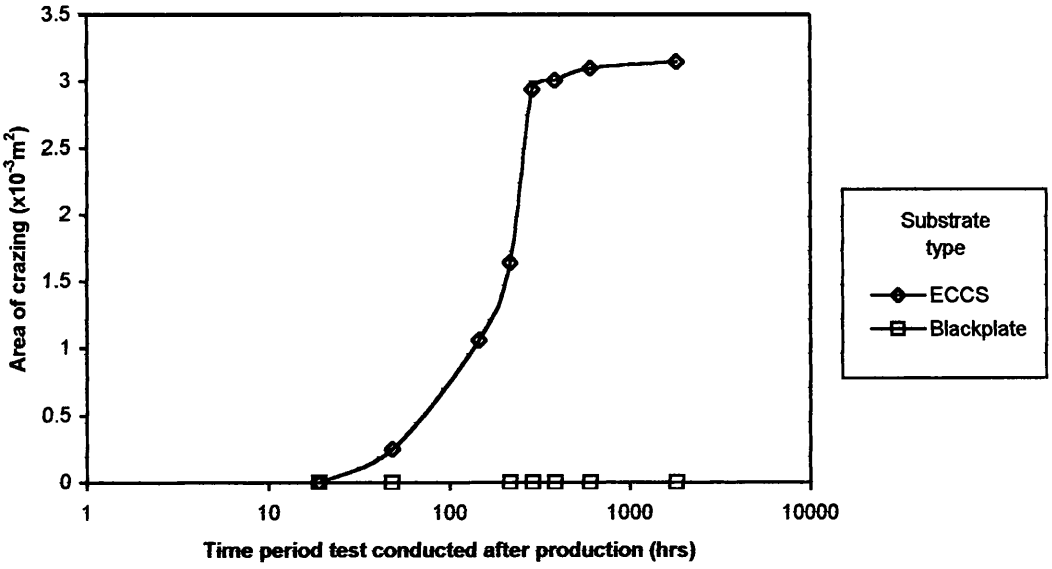

Figure 6.4. Comparison of blackplate and standard substrates on the craze susceptibility of film laminated materials.

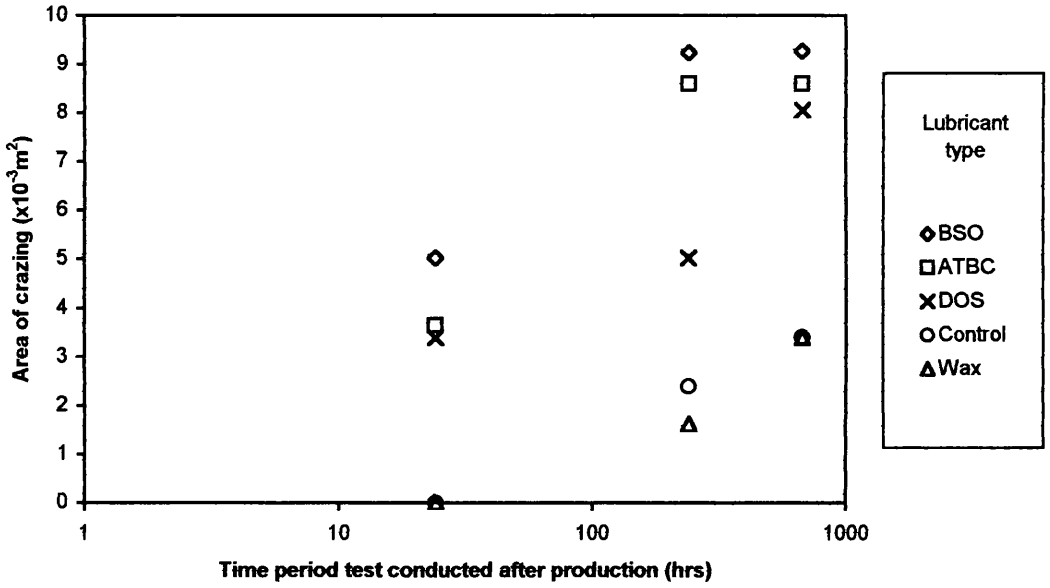

Figure 6.5. The effect of excess surface lubrication on the craze susceptibility of recently produced film laminated materials.

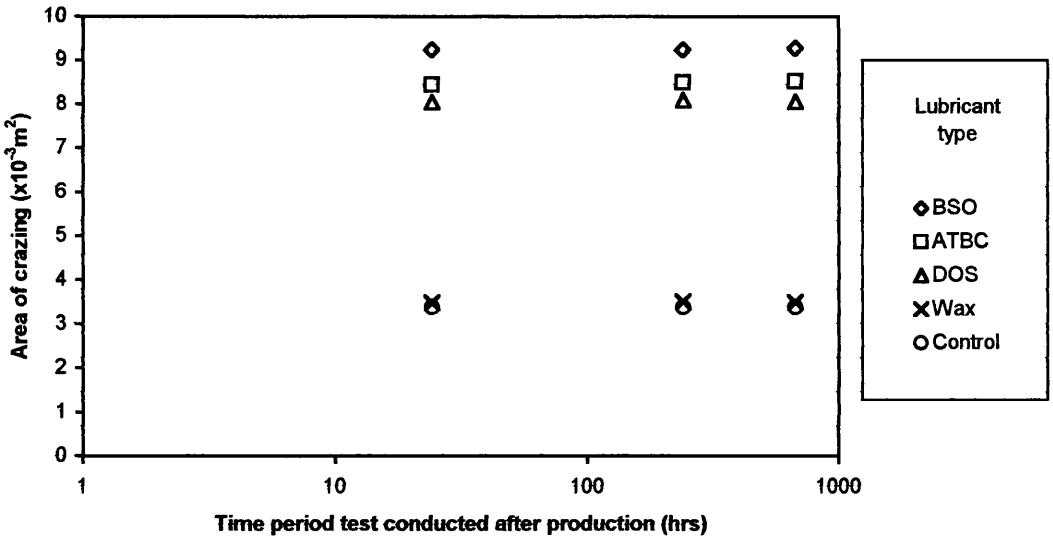

Figure 6.6. The effect of excess surface lubrication on the craze susceptibility of aged film laminated materials.

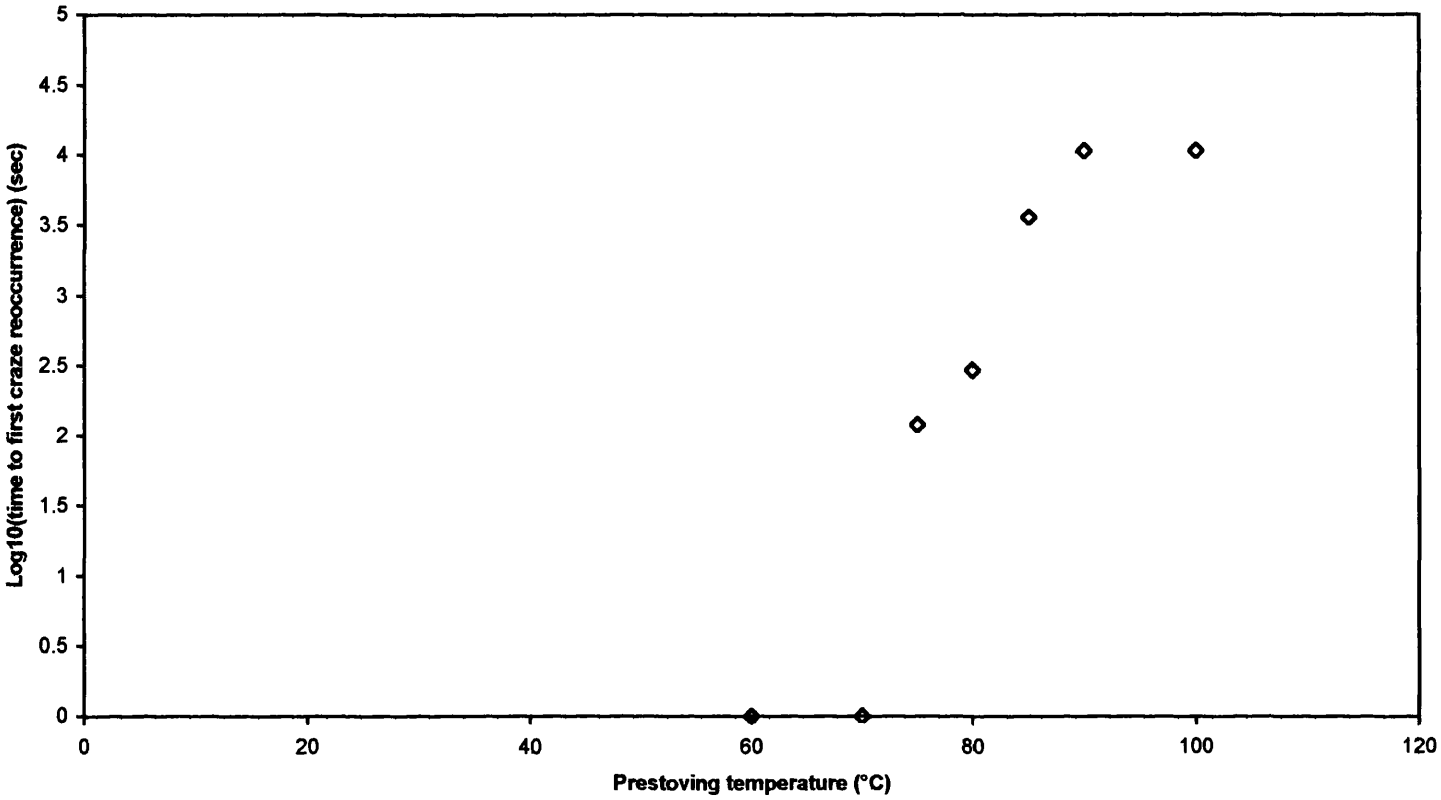

Figure 6.7. Time period after prestoving, which results in appearance of crazing in film laminated materials when biaxially stressed.

Appendix

Reoll & Korthauss cupping press operating procedure

The Roell and Korthaus press is a hydraulically activated cupping press for use with tin mill products. By producing cups it allows the evaluation of earring characteristics and consequently the effect of various defects on drawing e.g. gauge, bruises. There is provision to vary the clamping pressures and drawing speeds, as required for different materials. Trained staff as designated by the management should only fit the different tool combination.

A. A LIST OF KNOWN HAZARDS

- Electricity -440V
- High pressure hydraulic oil pipes could pose a problem if ruptured.
- Hydraulic oil if spilt on the floor could cause slipping. It is also an irritant.
- There are various pinch zones, mainly associated with breech and die.
- These areas should be avoided whilst there is power on or pressure in the system.

B. THE APPROVED SEQUENCE OF ACTIVITIES

Only trained and approved authorised operators are allowed to use this machine.

The majority of the controls are situated on the right hand side of the operator.

1. The mains power isolator (grey box) on the right hand side of the main unit should be switched "ON". The smaller switch above it should then be turned "ON" (vertical orientation).
2. The large red "EMERGENCY STOP" button (front right hand side) has a locking collet, which locks it in the "OFF" position once depressed. This should be freed by a 90^0 rotation of the collate ring. A red light adjacent to the emergency stop button should now be lit.
3. The red lever adjacent to the light and emergency stop should be set to its front facing position. This enables the lift off feature which in conjunction with the electronic control indicator is used to reduce the clamping pressure on the ears of the cup by cutting the pressure at the top of the stroke.

4. The breech mechanism holds the die and is a heavy item, provided with a counter balance to aid lifting it from the block. To open the breech, move the handle towards the operator. The handle on the left should be grasped in addition and the breech lifted upward clear of the block until it engages with the safety clasp.
5. The lubricated disc should now be placed on the blank holder and located centrally. It is advisable to use the locating ring for all disc gauges of .16mm and above.
6. The breech mechanism should now be lowered into position and locked by rotating the right hand lever away from the operator to take contact with the breech safety switch.
7. The R&K is now ready to operate. There are two operating speeds available, the normal speed using hydraulic pressure from the pump to activate the punch and high speed which uses an accumulator to vent the higher stored pressure to drive the punch.

Low Speed Operation

L1. Put high speed selection lever on left hand side of the accumulator to the "closed" position.
L2. Place small lever on top of accumulator to "normal"
L3. There are two selector knobs at the rear of the main unit, just above the horizontal surface. Both should be set to "HIGH RANGE".
L4. Turning it fully clockwise should close the dump valve, which is the valve on the bottom right on the front of the main unit.
L5. The clamping pressure valve located above the dump valve should also be closed, fully clockwise.
L6. Turning it fully anticlockwise should also close the left-hand valve labelled "testing speed" which controls the punch speed.
L7. The breech should be checked to ensure no loose matter is caught in it and that the handle is against the safety switch.
L8. Simultaneously press the right and left hand start buttons on the front panel. This necessitates the use of both hands and avoids the possibility of a hand

being in the pinch zones. The pump should start. If it does not then check the breech as described in L7.

L9. Set the clamping pressure in accordance with the material used by operating the clamping pressure valve to obtain the required pressure, which will be indicated on the rear lower, left hand dial, the high range being the outer red digits.

L10. Opening the "testing speed" control valve can now operate the punch. The speed of the punch can be controlled by the amount the valve is opened. A visual indication of the valve opening is provided by graduated markings 0 -10 on the valve control knob. The punch displacement is displayed on the lower of the three electronic control indicators and is labelled RAM. A linear gauge in the centre of the rear panel also indicates the displacement.

L11. At the end of the stroke the cup will be drawn through the die and knocked off the punch by the extractor rod. The punch will automatically retract and the pump will switch off with the punch fully retracted.

L12. At any stage in the drawing operation the punch movement can be halted by turning the testing speed control valve to "0". The test can be aborted at any stage by pressing the button labelled "stop" on the right hand side of the front panel. Pressing the "stop" button will drive both the outer and inner ram back to their fully retracted start positions. The outer ram supplies the clamping pressure and the inner ram the punch movement.

If at any time during the above testing procedure there is any malfunction of the machine the "EMERGENCY STOP" button must be pressed and advice sought from the trainer.

High Speed Operation

H1. Set high-speed lever to "OPEN".

H2. Set small lever to "HIGH SPEED" and secure with an elastic band attached to the pressure dial.

H3. There are two selector knobs at the rear of the main unit, just above the horizontal surface. Both should be set to "HIGH RANGE".

H4. Turning it fully clockwise should close the dump valve, which is the valve on the bottom right on the front of the main unit.

H5. The clamping pressure valve located above the dump valve should also be closed, fully clockwise.

H6. Turning it fully anticlockwise should also close the left-hand valve labelled "testing speed" which controls the punch speed.

H7. The breech should be checked to ensure no loose matter is caught in it and that the handle is against the safety switch.

H8. Simultaneously press the right and left hand start buttons on the front panel. This necessitates the use of both hands and avoids the possibility of a hand being in the pinch zones. The pump should start. If it does not then check the breech as described in H7.

H9. Set the required clamping pressure.

H10. Charge the accumulator by opening the "testing speed" valve. The dial on the accumulator cabinet will show the pressure charged.

H11. When 45 bar is attained shut off the "testing speed" valve. If the pressure were to exceed 50 bar a relief valve will automatically operate. Never operate the punch when this occurs as the fluctuations in pressure throughout the system can cause the clamping pressure to fall below a satisfactory level resulting in a badly wrinkled cup and possible die damage.

H12. Check the clamping pressure.

H13. When satisfied the unit is ready to operate activate the discharge by means of the "HS START" button on the near right hand side of the accumulator cabinet top.

H14. At the top of the stroke press, with the right hand, the green button labelled cup extractor" and keep it pressed until the punch and extractor starts to retract. As retraction commences remove the cup with the left hand. When retraction is complete the pump will switch off.

H15. On completion of cupping follow steps L1 to L6. Open the dump valve to relieve any stored pressure then close it.

H16. Put the small power switch at rear right hand side of the main unit to "OFF" (horizontal orientation) and finally switch of the main isolator.

If at any time during the above testing procedure there is any malfunction of the machine the "EMERGENCY STOP" button must be pressed and advice sought from the trainer.